

AD/A-006 548

PRODUCTION AND ENGINEERING METHODS
FOR CARB-TEK[®] BATTERIES IN FORK
LIFT TRUCKS

Joseph E. Metcalfe

Standard Oil Company (Ohio)

Prepared for:

Army Mobility Equipment Research and
Development Center

December 1974

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report describes the production of battery cells in the Sohio battery pilot line. A description of process improvements studies on various cell components is given. Pictures showing facilities, cell components and completed cells is presented.			

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SUMMARY

The objective of this contract was to reconstruct the Sohio battery pilot line and begin the studies on production and engineering methods for the production of Carb-Tek® batteries.

The battery pilot line has been refurbished and returned to working order. This work was mostly mechanical construction.

Process improvements for anodes, cathodes and separators have been made. From this work it is projected that sealed battery cells producing 25 watt hours/lb will be achievable.

Pictures showing the pilot facilities as well as sealed battery cell components and sealed cells are presented.

PREFACE

This interim report was prepared by the Research and Development Department of The Standard Oil Company (Ohio) called hereinafter Sohio under U.S. Army Mobility Equipment Research and Development Center (USAMERDC) Contract No. DAAK02-73-C-0493.

The main purpose of this contract was to reconstruct the Sohio battery pilot line facility. This facility is needed to produce battery cells and develop production and engineering methods for the Carb-Tek[®] battery. The ultimate goal of demonstrating a molten salt battery in a fork lift truck application is conceived to be a three year effort.

Acknowledgement is made to Mr. Glenn Abrams for his contributions to the reconstruction of the battery pilot line. Many thanks to Mr. James C. Schaefer and his ESB, Inc., personnel for their aid during the course of this work and the preparation of this report.

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INTRODUCTION

The ultimate goal of this effort is to develop the manufacturing technology of the Carb-Tek® battery to such a point that this type of a battery can be inexpensively fabricated in production quantities to meet military specifications. It is believed that the necessary manufacturing technology can be developed during this technical effort time. At the end of the technical effort a prototype fork lift truck battery will be delivered for testing. The proposed specifications of the battery are as follows:

Nominal Voltage - 36 volts at full charge
30 volts at cut-off

Delivered Capacity - 720 amp hours when discharged
from full charge to cut-off voltage

Recharge Rate - 1 hour to restore 80% of capacity

Weight - Less than 1550 lbs.

Est. Volume - 9.25 cubic feet

Objective:

The objective of the first year's work was to reconstruct the Sohio battery pilot line and begin the studies on production and engineering methods for the production of Carb-Tek® batteries. The work accomplished during the first year will serve as a foundation for the fabrication of modules and batteries during the succeeding years.

An additional objective, the involvement of a battery company in this work, had been undertaken. Sohio has negotiated with ESB Inc. to actively participate in this contract. The participation of a knowledgeable battery manufacturer in the early stages of the program, should enhance the probability that a battery manufacturer will be prepared to manufacture the Carb-Tek® battery upon the successful completion of this technical program. Of course the government will be supplied detailed procedures for all manufacturing stages of the battery during the course of this effort and may seek out other manufacturers to produce the Carb-Tek® battery for government uses.

Background:

In the late 50's Sohio embarked upon fuel cell research which was the forerunner of the Sohio Carb-Tek® battery program. Research on fused salt batteries started in 1962. During the period of 1962-1971, Sohio built up considerable patent and know-how position in the area of molten salt batteries. During this 10-year period of time the Carb-Tek® battery was developed.

The Carb-Tek® battery employs a porous carbon cathode, a lithium-aluminum alloy anode, a eutectic mixture of potassium chloride and lithium chloride

as the electrolyte, and a tellurium tetrachloride additive. The porous carbon cathode is an aggregation of active carbon particles which have been pressed together with a suitable binder. The active carbon particles can be considered as microscopic crystallites with graphite-like properties which are bonded or cross-linked together. The porous carbon itself contributes to the capacity by providing literally acres of surface area for energy storage using the Helmholtz double-layer principle. However, after treating the carbon with the tellurium tetrachloride additive, the carbon contribution over the voltage range of interest (3.25 volts to 2.5 volts) is negligible.

The Carb-Tek[®] battery employs a solid plate anode which is an alloy of aluminum and lithium. The use of this anode avoids corrosion and other problems associated with operating with liquid lithium anodes. This electrode operates over a range of lithium composition from about 10 mole percent to nearly 50 mole percent and develops a porosity as it operates. Fully conditioned anodes will operate at current densities up to 2 amps/cm² without serious polarization, and will operate at substantially higher currents for power bursts of short duration (1-2 seconds). Use of this electrode reduces the cell potential by about 0.3 volt compared to the pure lithium anode.

The tellurium tetrachloride is chemisorbed into the cathode with the bonding probably occurring at the edge of the microcrystallite as shown in Figure 1.

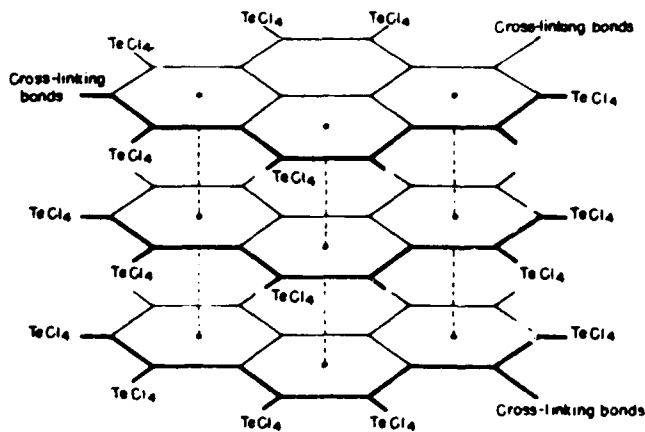


Figure 1 - Microcrystallite of Active Carbon Reacted with TeCl₄

The tellurium tetrachloride undergoes the following reactions and produces the indicated electromotive forces (EMF).

Reaction	EMF (Volts)
(1) $4 \text{ Li} + \text{TeCl}_4 \longrightarrow 4 \text{ LiCl} + \text{Te}$	3.1
(2) $\text{Li} + \text{Te} \longrightarrow \text{LiTe}$	1.75

Theoretical energy densities for this system are:

System	Theoretical Energy Density (Watt-Hours/Pound)
$4 \text{ Li} + \text{TeCl}_4 \longrightarrow 4 \text{ LiCl} + \text{Te}$	510
$5 \text{ Li} + \text{TeCl}_4 \longrightarrow 4 \text{ LiCl} + \text{LiTe}$	575

While $\text{Li} + \text{Te}$ can theoretically react to produce Li_2Te there is no evidence in any published work that the reaction proceeds beyond LiTe . If techniques are developed to react completely to Li_2Te , the limiting theoretical energy density of this system would be in excess of those previously stated.

The tellurium tetrachloride additive has a reaction plateau at about 2.75 volts which should permit a reasonably simple voltage control system. Typical cell discharge curves at the one C rate are shown in Figure 2.

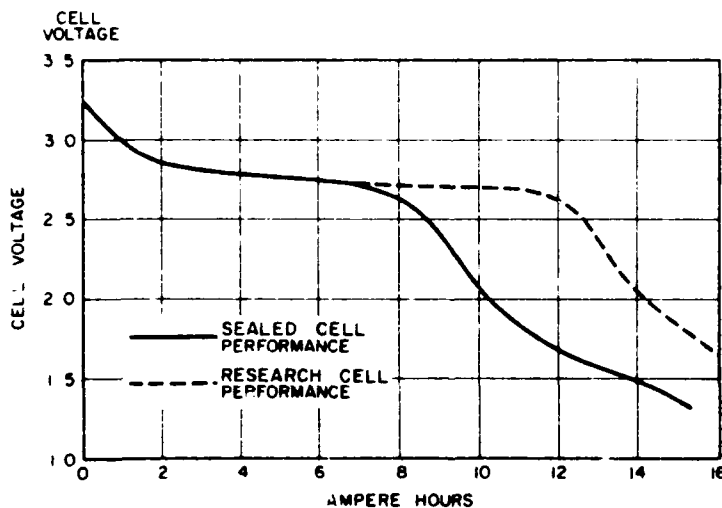


Figure 2 - Cell Voltage vs. Amp-Hours Current Draw

In Figure 2, the solid line depicts the discharge curve which has been demonstrated in sealed cells measuring 6" x 4-9/32" x 21/32". These cells have demonstrated an energy density of 25 watt hours per pound when discharged from 3.25 volts to 2.5 volts (discharge to 1.0 volts has demonstrated an energy density of 40 watt hours per pound).

A higher energy capacity per unit volume of carbon electrode has been achieved with research size electrodes (1-1/2" x 1/2" x 1/8") than has been achieved in sealed cells. If this electrode performance can be obtained in a sealed cell, an energy density of 35 watt hours per pound can be obtained. The broken line in Figure 2 depicts the performance achieved on a research size electrode scaled up to a sealed cell electrode size.

Power densities in excess of 200 watts per pound have been demonstrated in tellurium containing cells of the type aggregated into batteries.

A cycle life of 1500 cycles has been demonstrated on carbon cathode cells but only 100 cycles on a 12 cell battery with tellurium treated cathodes.

In 1971 a decision was made in Sohio to terminate all efforts in this research area until market needs and a viable business plan were developed. At this time the Sohio battery pilot line and research facilities were put into storage.

INVESTIGATION

Most of the effort on this contract has been devoted to reconstructing the battery development facilities and familiarizing the ESB, Inc., personnel with the Carb-Tek[®] battery technology. In addition to the main tasks, studies have been conducted to improve process conditions for anodes, cathodes and other cell components and to determine the size of cell needed for a fork lift truck battery application.

Pilot Line Facility

The reconstruction of the facilities has been reported on previously, and photos of many pieces of equipment have been shown (1). In Figure 3 is shown a block diagram of the facility. Each inert atmosphere box (1,2,3,4) is equipped with its own purification system and is isolated via a bulkhead door system 9. Materials are brought into the inert atmosphere via entry port 5. Box 3 is used primarily for materials receiving and storage. Boxes 1 and 2 are used for battery cell assembling. Box 4 is used for electrochemical formation of the carbon cathodes in the formation tank 6. The dump tank-loader 8 is used to supply electrolyte for the electrochemical formation via the heated pipe 7.

The inert atmosphere facility used for anode and cathode process studies is similar to one section of the main engineering facility (1).

The original inert atmosphere boxes and gas purification systems were supplied by Vacuum Atmosphere Corporation of Hawthorne, California. During prior operation and during the reconstruction several modifications were made to the purification systems (Model No. HE373) and associated plumbing (Figure 4). The major changes were as follows:

- (1) Larger diameter copper tubing (1-1/2 inch) was used to minimize pressure drops. In previous work it had been found that excessive pressure drops were the main cause of failures in the circulating blowers.

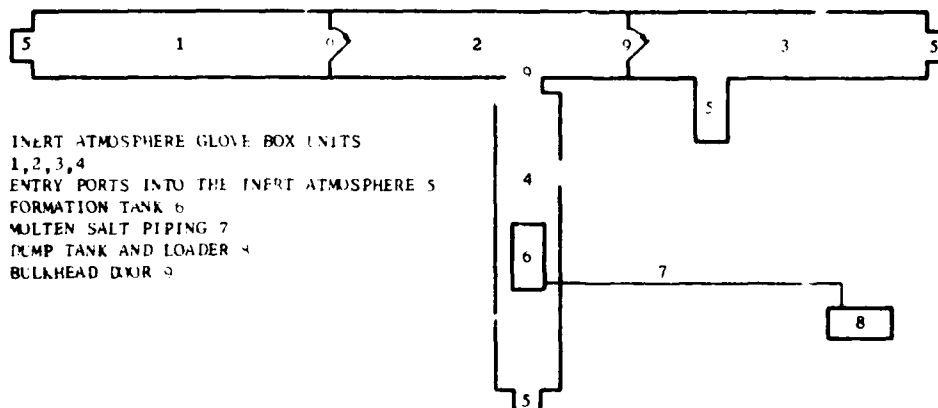


Figure 3 - Block Diagram of Carb-Tek[®] Battery Facility

- (2) A differential pressure gauge, Model 2080 from F. W. Dwyer Manufacturing Co., was installed to monitor the pressure drop across each circulation blower.
- (3) Dual molecular sieve towers were installed in parallel for drying the argon and assisting in minimizing the pressure drop across the system.
- (4) The original titanium getter furnace in the system was replaced with a Sohio designed system. The new system eliminates both heater failures and reactor basket distortion which plagued the original Vacuum Atmosphere getter furnace. This unit is operated at a temperature which would be safe for the lowest melting binary alloy that could possibly form in the total system.
- (5) The mechanical switch for automatic pressure control was replaced with an F. W. Dwyer "Photohelic" pressure switch/gauge, Model 3310 to control the pressure of the dry box atmosphere. The dry box is connected to the low pressure input to provide better isolation from the atmosphere and to eliminate a possible leak.

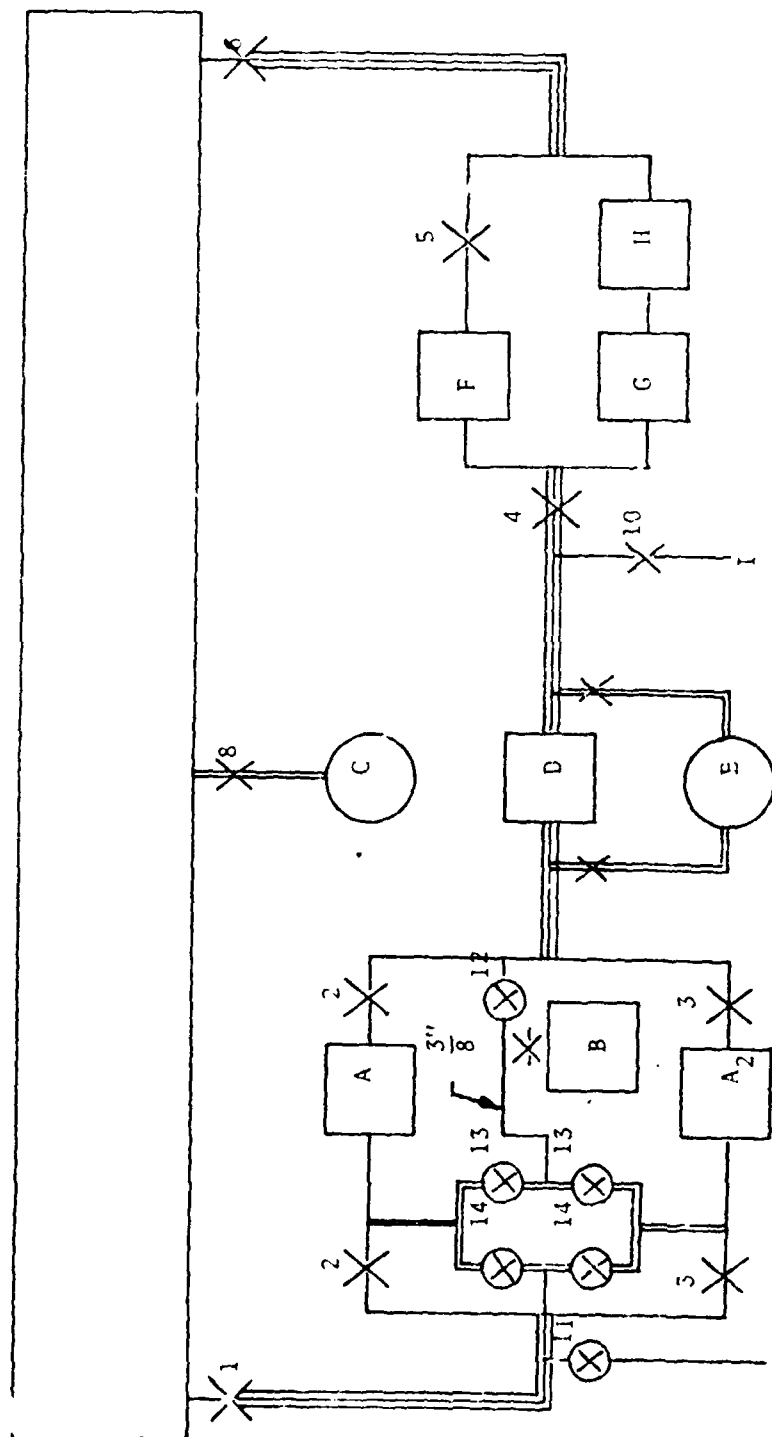
As each section of the inert atmosphere box and purification system was completed, they were checked for leaks with a helium mass spectrograph leak detector (Consolidated Electronics Model 24-120B). The dry train was connected to the leak detector after isolation and a vacuum was drawn on the complete system. Helium was then sprayed around all connections, valves and other components of the dry train. A leak rate of 3.1×10^{-10} atmosphere cc/sec can be determined by this instrument with this procedure. The second step in the leak detection was to isolate the dry box and fill it with helium. Using the helium mass spectrograph type instrument with a sample probe "sniffer", a leak rate of 10^{-4} atmosphere cc/sec can be determined.

The dry train was then completely hooked up to the dry train nitrogen getter furnace. Helium was then allowed to fill all components of the dry train and dry box system. The helium mass spectrograph leak detector was used on all components of the system.

The completed system was then purged completely with argon for approximately 30 times. This diluted the air in the dry box system sufficiently to start the purification system. The nitrogen getter was activated by controlling the retort wall temperature at 1600°F with the system evacuated for 16 hours. The argon was then allowed to circulate throughout the box, purification system, and the nitrogen getter furnace.

The quality of the inert atmosphere system was monitored by several methods. Moisture was continuously monitored with an aluminum oxide hygrometer manufactured by Panametrics, Inc. This unit, a model 1000, is capable of measuring dew points from +20°C to -110°C when equipped with the standard sensor probe. Nitrogen was determined by using a Varian Associates trace gas analyzer, series 1532, equipped with a 24 ft. long, 1/8 in. dia. 5A molecular sieve column. The trace gas analyzer operates with a sensitivity of about 0.50 millivolt signal for 1 ppm nitrogen in a 1 ml sample of argon.

THE CONVERTED DRI-TRAIN SYSTEM



Argon Inlet

- A Drying Towers
- B Vacuum Pump
- C Photohelic
- D Rotron Blower
- E Magnehelic
- F 1-heat exchanger
- G Nitrogen Getter
- H 2-heat exchangers
- I Leak Check Port

- 1 Box Outlet Valve
- 2 Isolation Valves - Dryer 1
- 3 Isolation Valves - Dryer 2
- 4 N₂ Getter Isolation Valve
- 5 Bypass Control Valve
- 6 Box Inlet Valve
- 7 Vacuum Pump Isolation Valve
- 8 Photohelic Isolation Valve
- 9 Magnehelic Isolation Valves
- 10 Lead Check Valve

- 11 Argon Inlet Valve
- 12 Argon Exhaust Valve
- 13 Dryer Evacuation Valve
- 14 Dryer Equalization Valve

Figure 4

Presently, several on-line oxygen monitors are being considered. A Research, Inc., Model 916 monitor has been used on the dry box system. Problems with instrument leaks and the ppm range extender resulted in erroneous readings on an atmosphere of very low oxygen content. An estimate of the oxygen concentration based on the nitrogen concentration would be less than .3 ppm. The overall quality of the atmosphere can be determined by removing parts of the envelope of a light bulb and determining the life in the atmosphere. Previous work by Sohio has indicated a life of approximately 60 hours corresponds to 1 ppm oxygen. In the dry box system bulbs have frequently operated over 3 weeks and some up to 10 weeks.

Cell Production

In Figure 5 is a material flow diagram for the production of molten salt battery cells. Each material is processed prior to the final assembly of the cell. In order to minimize cell contamination, the assembly takes place in the inert environment. A schematic representation of a cell is shown in Figure 6. A description of each cell component and its associated processing follows.

Cathodes

The carbon cathodes used in the pilot line work consists of two carbon plates made from activated carbon supplied by Pittsburgh Activated Carbon Co. (PACCO) and fabricated by the Pure Carbon Co. In order to minimize the voltage drop throughout the cathodes, a special current collector is sandwiched between the carbon plates. This special current collector consists of two 5 mil tungsten flat strips laminated between two sheets of Grafoil®. This current collector was supplied by Union Carbide Corp. The other major part of the cathode assembly is a graphite connector which serves as a termination point for the Grafoil® current collector and the tungsten rod which passes through the cell seal to provide the electrical connection (Figure 6). Union Carbide Corp. Carbon Cement type 140 was used to glue all carbon parts. The machining of the carbon parts to size and final assembly was provided by a local carbon fabrication shop. (Gaines Industries)

The cathode baking and TeCl_4 impregnation flow diagram is shown in Figure 7. The fabricated cathodes were first inspected and weighed. They were then placed in a reactor and heated for 6 hours at 600°C in order to cure the carbon cement. During the curing process the reactor was purged with argon. The temperature of the reactor was increased to 900°C and the reactor placed under a vacuum for 13 hours in order to remove adsorbed gases from the carbon. The reactor was then cooled to room temperature and taken into the dry box for opening. The cathodes were then weighed and inspected again inside the dry box. The cathodes were then loaded into the impregnation vessel with a weighed amount of tellurium tetrachloride. The vessel was heated to above the vaporization temperature of the tellurium tetrachloride (450°C) to allow the carbon cathodes to adsorb the vapor. After 4 hours of exposure to the TeCl_4 vapor the reactor was cooled and introduced into the inert atmosphere dry box for unloading.

MATERIAL FLOW DIAGRAM

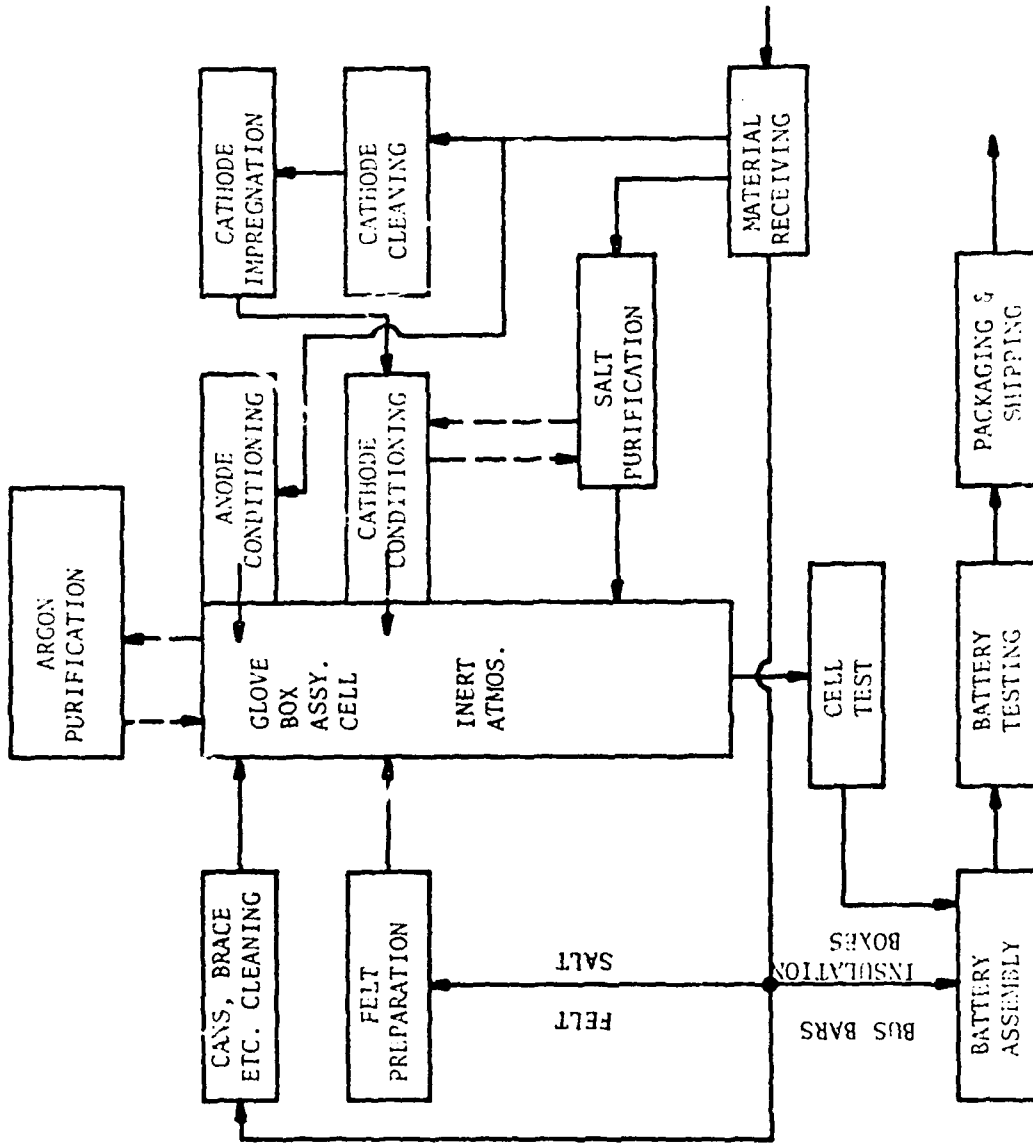


Figure 5

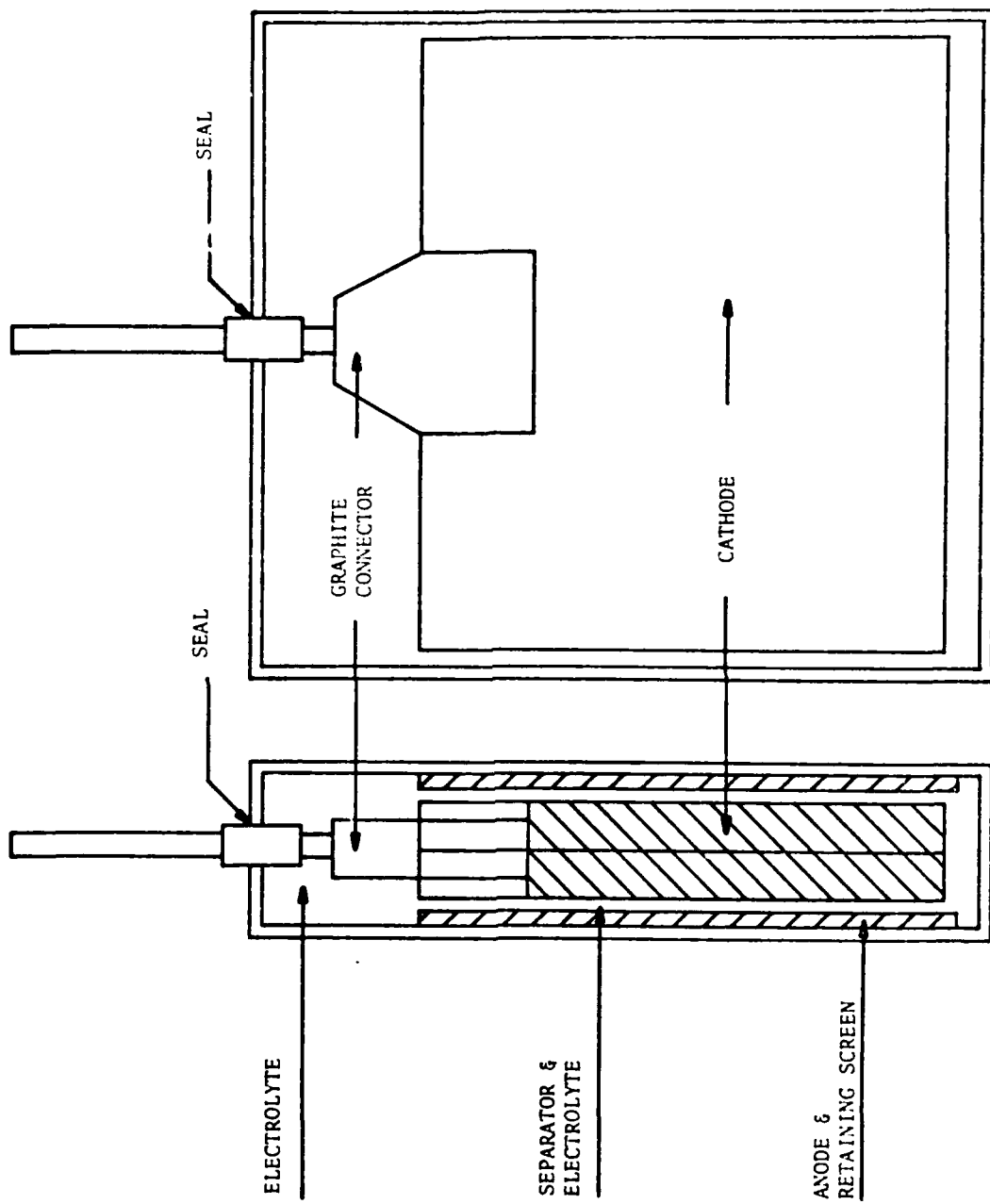


Figure 6

CATHODE FLOW DIAGRAM

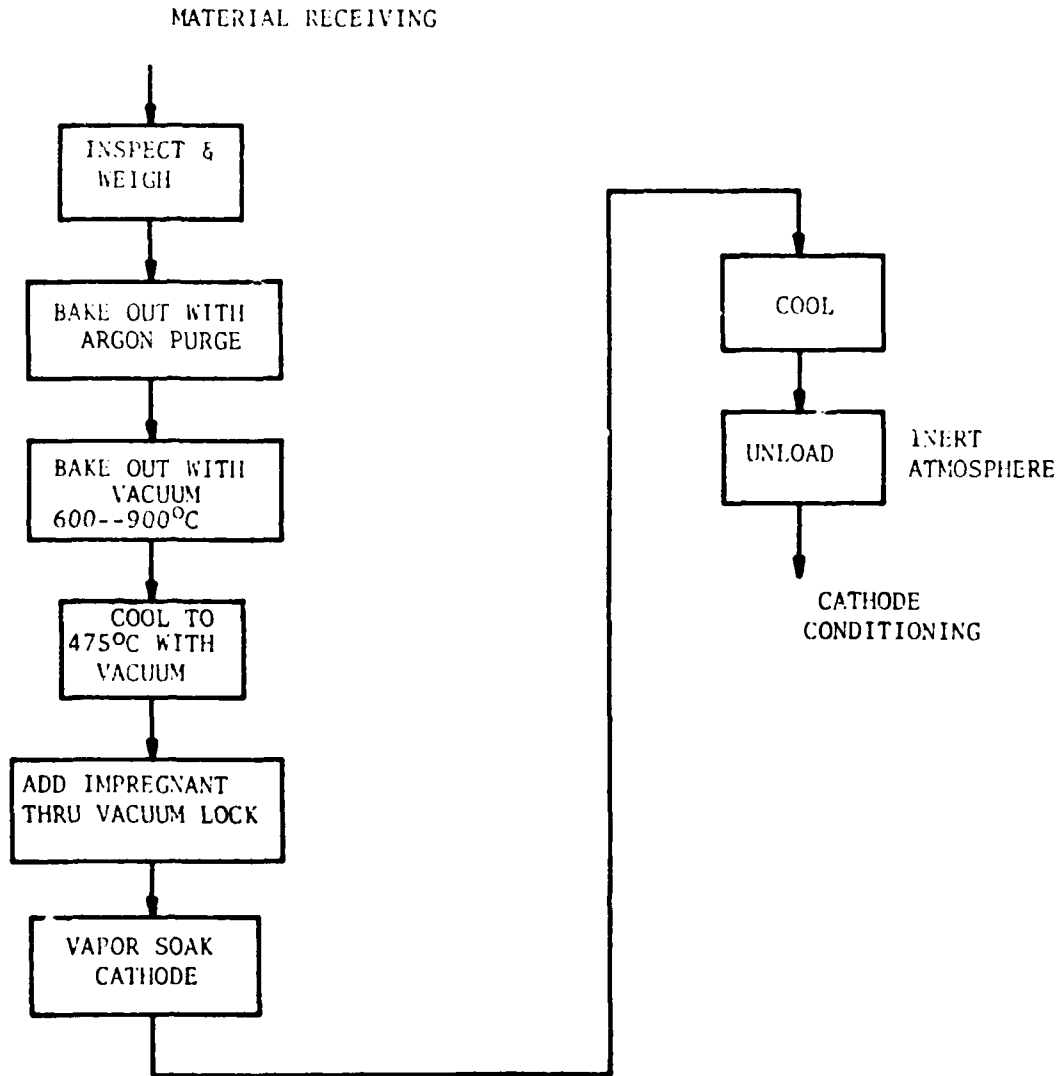


Figure 7

purposes. The cathodes were weighed again and inspected. Approximately 90% of the TeCl_4 was adsorbed by the carbon. The cathodes were then ready for the cathode conditioning step.

As shown in the cathode conditioning flow diagram, Figure 8, a tungsten rod conductor was inserted into the graphite connector of the carbon cathode. This was done by clamping the graphite connector between two carbon plate resistive heaters which were connected to a power supply. The graphite connector was slowly heated to 500°C at which temperature the tungsten rod was inserted. After cooling the cathode was ready for conditioning in the formation tanks.

The formation tank (Figure 9) is a heated vessel containing equally spaced sacrificial anodes of lithium-aluminum which are immersed in molten salts. The tank can be filled or emptied through connections to a molten salt dump tank via a heated stainless steel piping system (Figure 3). Molten salt is transferred back and forth from the formation tank to the dump tank using pressure and vacuum. The cathodes were attached to the lid of the formation tank and inserted into the formation tank. They were positioned between the sacrificial anodes. The cathodes were then electrically connected to a power supply for automatic charging and discharging (Figure 10). During this step the carbon cathode undergoes molecular rearrangement giving off a variety of carbon-hydrogen, oxygen, chlorine compounds as well as electrochemically forming the Te-Cl complex on the carbon surface. Before initiating the cycling the tank was filled with electrolyte. The salt tank was then evacuated and pressurized several times to insure wetting of the carbon cathodes. The electrodes were automatically charged and discharged from approximately 3.35 volts to 0.8 volts. Upon completion of approximately 15 charge-discharge cycle, the cathodes were removed from the salt tank and permitted to cool. During the course of this cycling the rate was varied from a 16 hour cycle to a .3 hour cycle. After cooling the cathodes were once again inspected.

Anodes

The lithium-aluminum anodes developed by Sohio provides the means of obtaining a high cell capacity and voltage without the difficulties associated with liquid lithium. Since lithium is highly reactive, it is difficult to prepare the lithium-aluminum alloy without introducing oxides and nitrides. There are two basic methods that are presently useful in the production of anodes: (1) Conventional metallurgy in which the lithium and aluminum are melted and alloyed in the proper proportions and then cast into the required shape, (2) Electrochemically "pumping up" aluminum to form a lithium-aluminum anode of the proper alloy content. Anodes produced by conventional metallurgy have been supplied by Foote Mineral Corp.

"Pumping up" aluminum currently appears to be the most expeditious method of producing anodes of the required purity. This method allows production of the anode in the inert environment of a sealed cell. Cells of

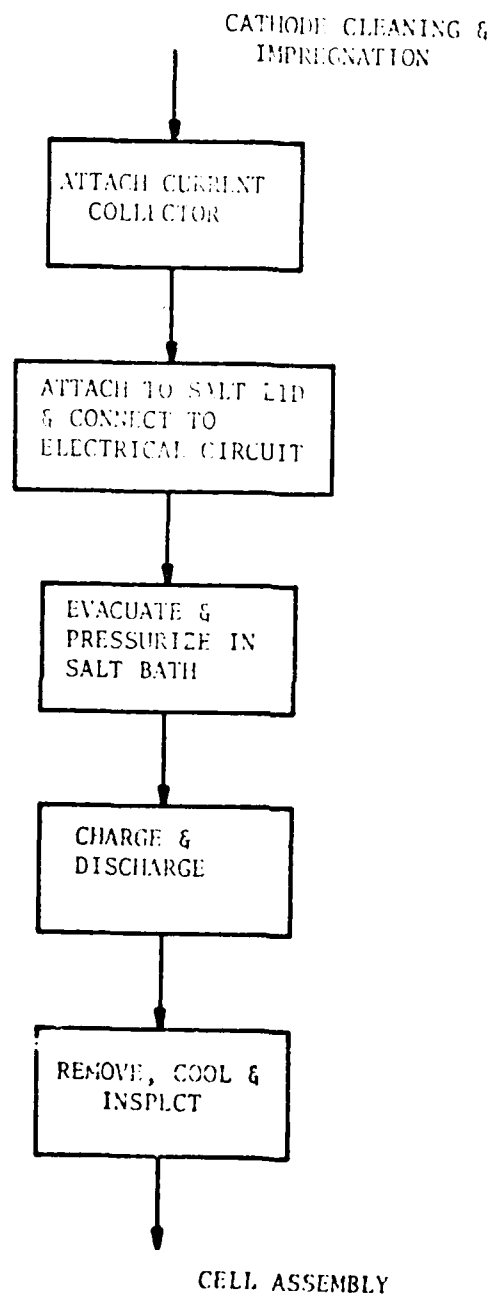


Figure 8

FORMATION
TANK



Figure 9 - Dry Box With Formation Tank

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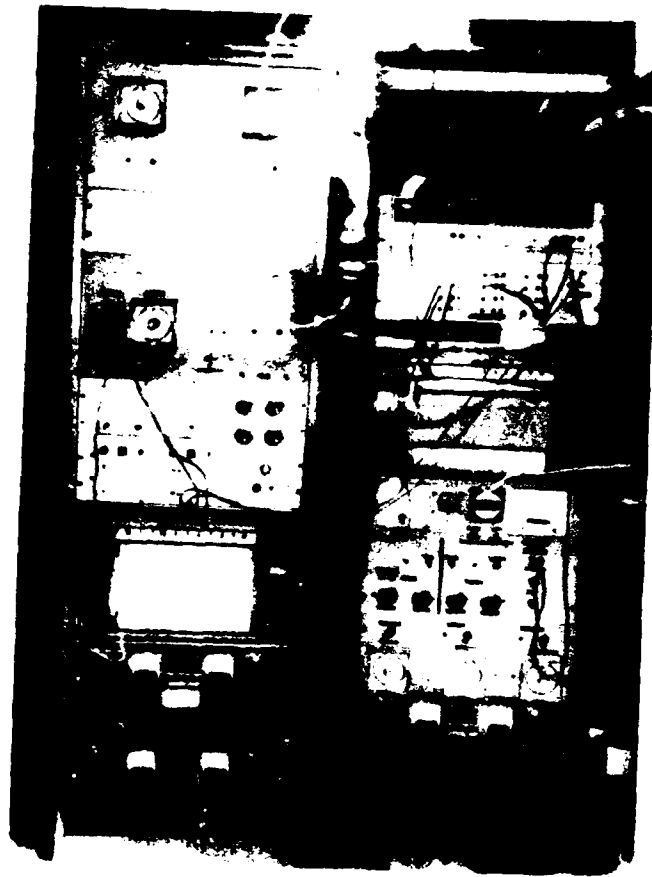


Figure 10 - Electronic Cycling
Equipment for Cathode Formation

this type were constructed using capacitor grade aluminum as the anode. The anodes were cut to size from 13 mil capacitor grade 99.96% (min) aluminum sheet and introduced into the dry box environment. The anodes were polished to remove the external oxide layer and weighed. The aluminum was then ready for assembly into the individual cells. These cells had a thicker cathode to compensate for the smaller volume of the aluminum anodes vs. the lithium-aluminum anodes supplied by the Foote Mineral Corp. Since the lithium content of a lithium-aluminum anode cannot be discharged below approximately 6 weight percent lithium during cell operations, the cathodes must be removed from the formation tank in a lower state of discharge than will be normally encountered during cell operation. Similarly the lithium contents of the electrolyte must be adjusted to be sure the lithium content of the salt mixture does not deviate too far from the eutectic mixture. As can be seen in Figure 1, the lithium content must be maintained between 30 and 47 mole % at 450°C. Failure to adjust the lithium content could result in the precipitation of one of the salts.

Separator

As shown by the felt preparation flow diagram, Figure 12, the boron nitride roving purchased from the Carborundum Company is used for the preparation of the separator material. The roving is weighed and boiled in a solution of water and methanol for 15 minutes to remove oxide impurities. The mixture is then decanted. The roving is then chopped into approximately one inch lengths in a blender with a distilled water solution. The blended mixture is diluted with distilled water and agitated with air in order to produce a homogeneous suspension. The suspension is vacuum filtered to form a damp mat. The mat is formed about a teflon mold sized to the shape of the cathode. The formed mat is then sprayed with a binding mixture made from the eutectic salt and water. The felt separator and mold are then air dried in an oven to remove the excess moisture.

After drying the boron nitride felt separator, the mold is removed and the molded separator is introduced into a vacuum bake-out chamber attached to the inert atmosphere dry box. In the vacuum bake-out chamber, the felt is heated to a 120°C and evacuated with a mechanical vacuum pump for 4 hours, after which, the temperature is then raised to 300°C and evacuated for an additional 10 hours. The vacuum chamber is then allowed to cool while maintaining a vacuum with a diffusion pump for 3 hours. The dried felts are then introduced into the inert atmosphere (without exposure to the laboratory atmosphere) and are ready for cell assembly.

Referring to the parts cleaning flow diagram, Figure 13, metallic parts (cans, lids, screens, and braces) are washed in trichloroethylene in an ultrasonic cleaner for approximately 1 hour and then rinsed in acetone followed by a rinse in distilled water. This is primarily a degreasing operation. The metal parts are then soaked in a dilute HCL bath to which Rodine has been added followed by a rinse in water. The purpose of this step is to remove any oxide films and to prevent any further oxidation of the surfaces prior to entry into the inert atmosphere. The parts are

LiCl-KCl PHASE DIAGRAM

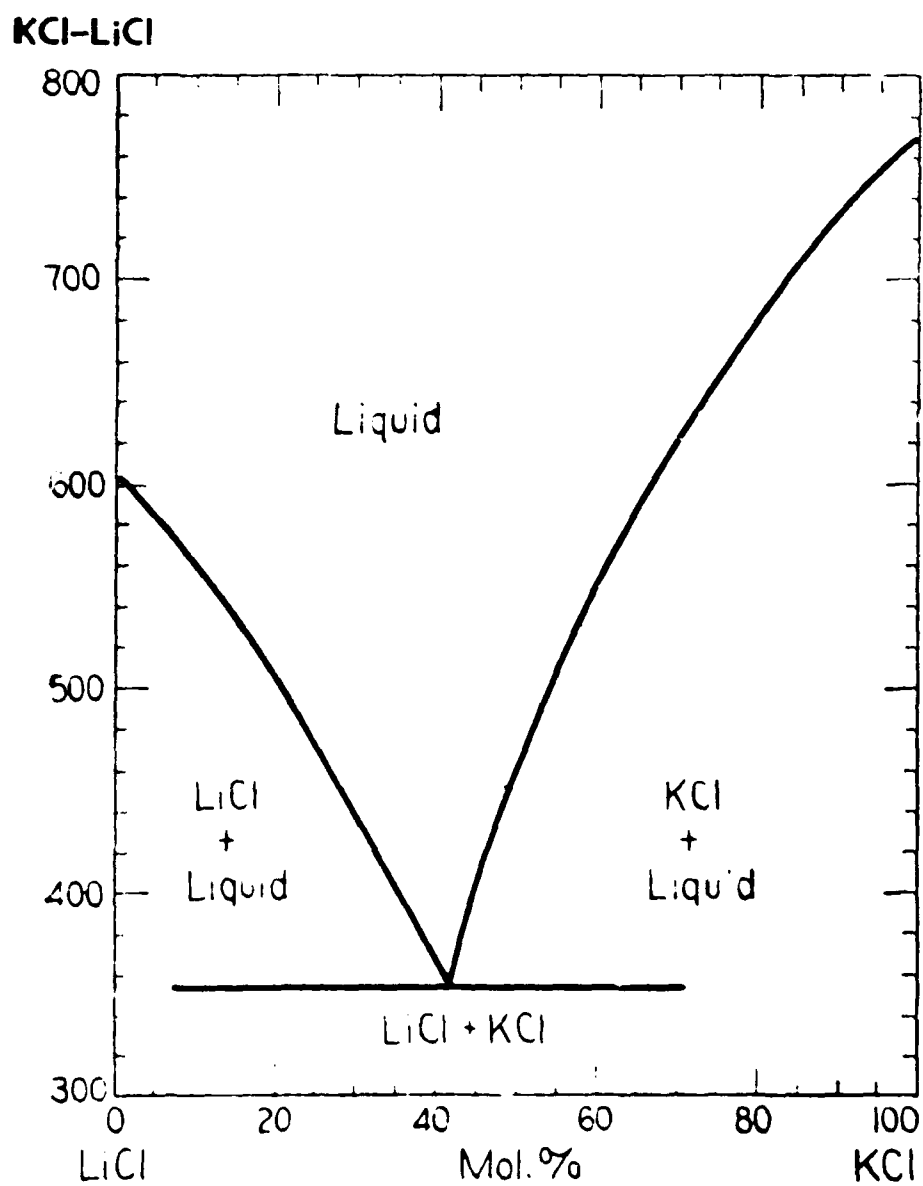


Figure 11

FELT PREPARATION FLOW DIAGRAM

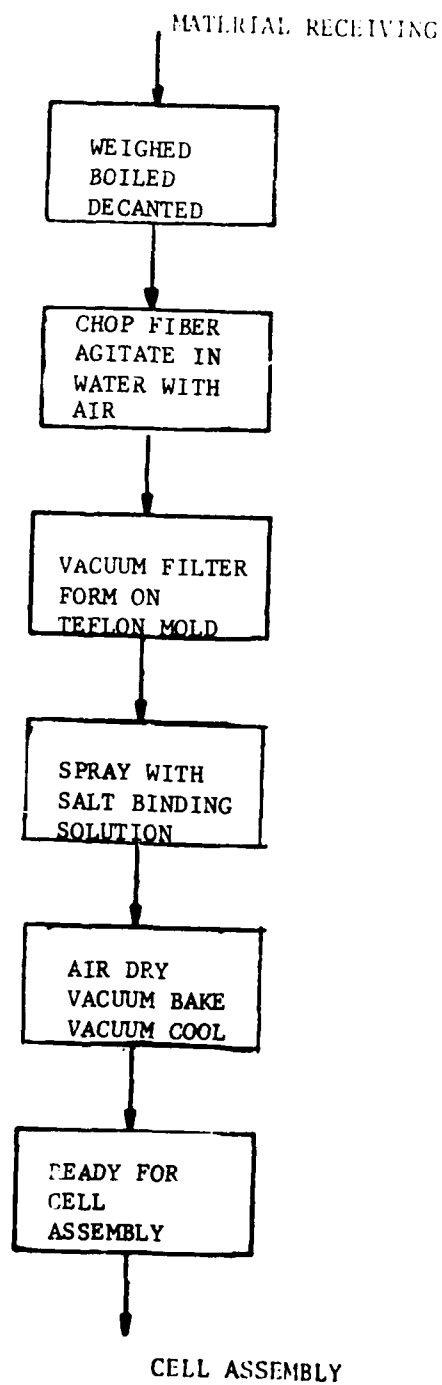


Figure 12

PARTS CLEANING FLOW DIAGRAM
(CANS, LIDS, SCREENS, BRACES)

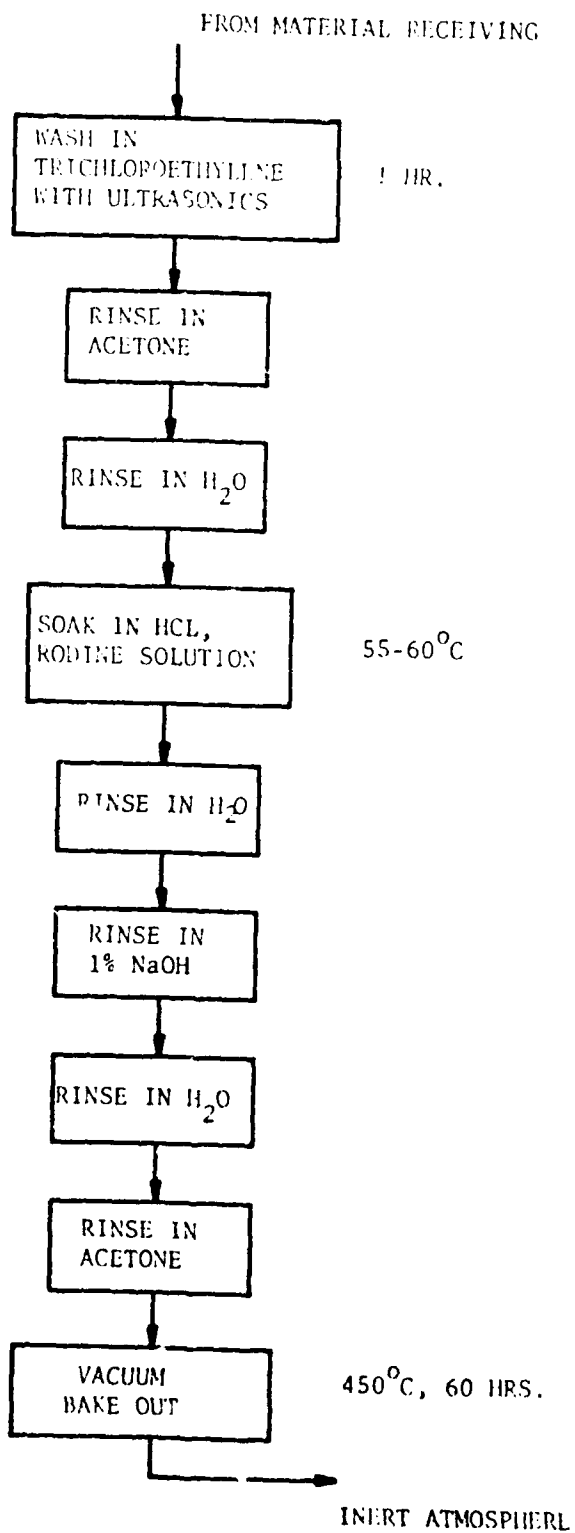


Figure 13

then rinsed in a weak sodium hydroxide bath followed by a water and acetone rinse to insure neutralization of the materials on the surface and to prevent further oxidation. After the rinse, the parts are inserted into a vacuum bake-out furnace attached to the dry box system. Under vacuum (with a diffusion pump operating) they are heated to 450°C and retained at that temperature in the furnace for 60 hours. After the bake-out period, the parts are transferred into the dry box assembly system for assembly of the cell.

Cell Assembly

The cell assembly flow diagram is shown in Figure 14. The cans are first welded in the dry box (Figure 15). The can (without the top) is then helium leak checked for poor welds. The cathode is then slipped into the molded boron nitride separator and in turn slipped into the screen basket (Figure 16). The boron nitride separator is constructed of two layers with alternating butt joints to prevent shorting at this seam. The separator is made taller than the cathode to permit folding of the separator across the top of the cathode. The basket assembly is slid into the can and the anodes are forced into the cans between the inside of the can and the screen basket. (Figure 6). The screen prevents a migration of anode material into the separator. The screen is spot welded to the can at the top. The area above the cathode is then packed with loose boron nitride felt. The spacers (Figure 17) are spot welded into the top of the can to prevent collapse and shorting of the cell when the can is subjected to vacuum. The lid is then welded to the top of the can. The electrolyte reservoir (containing powdered LiCl-KCl salt), anode conductor and the cathode current collector tube are welded to their respective connections, (Figure 15). The cold seal is then fitted (Figure 17). This is an insulated fitting containing a viton O ring and is supplied by Conax Corp. (Figure 17). This complete assembly is helium leak checked to insure good welds and connections.

The cells (sealed via a valve) are then removed from the inert atmosphere box. (Figure 18) The cells are then placed in a furnace at the cell conditioning station and connected to a vacuum system via the valves on the salt reservoir (Figure 19). The cells are then brought to temperature (500°C). The salt in the reservoir is then melted and allowed to fill the cell. Alternate pressure-vacuum pulses are used to insure filling of the cell. The cell is then subjected to several charge-discharge cycles to condition the anode as well as removing any impurities that are generated from the electrochemical cycling. Vacuum pulses are applied to aid in the removal of trapped gas impurities. After this conditioning period the cell is brought to full charge (3.30 volts) and the reservoir removed. A completed cell ready for further cell testing is shown in Figure 18.

Analysis and Optimization of the Process Steps

In order to expedite the development of improved cells for the fork lift truck application, parallel studies have been undertaken to improve the

CELL ASSEMBLY FLOW DIAGRAM
Inert Atmosphere

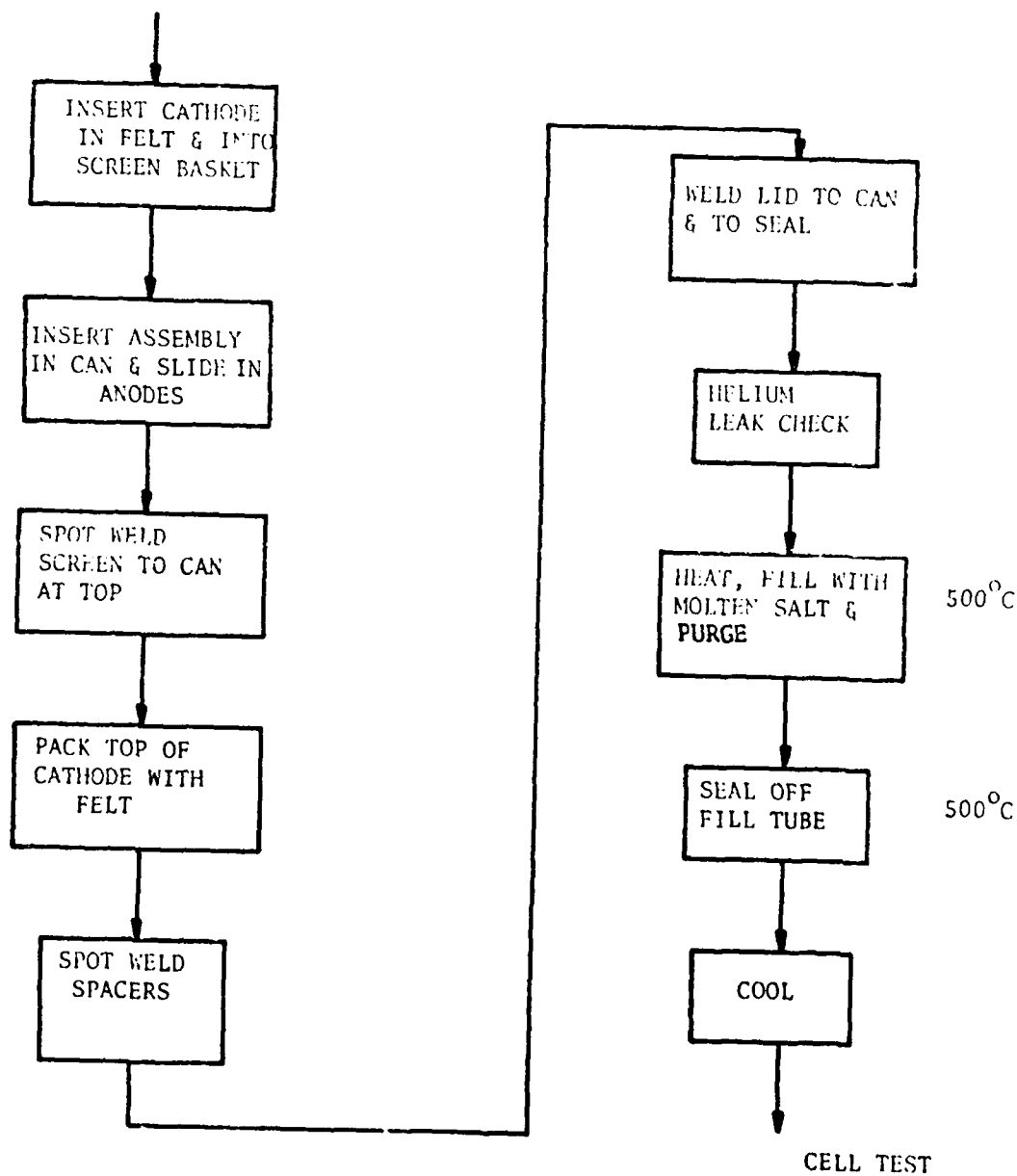
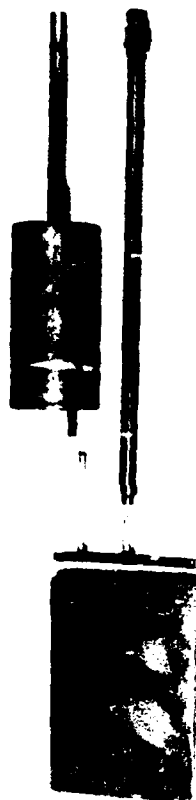


Figure 14

Salt
Reservoir



Cold Zone Seal

Cell Lid

Cell Can

Figure 15 - Cell Parts Before Welding

Boron
Nitride
Felt

Cathode
with
Tungsten
Rod



Anode

Screen
Basket

Figure 16 - Cell Components Prior to Assembly

cell components during the reconstruction period of the pilot line facility. This information will be used in the future cell designing-construction work that will take place in the pilot line facility. A description of the work on the various cell component-processes follows:

Carbon Cathodes

During the years that Sohio was developing the molten salt battery many types of Activated Carbons were tested as potential cathode materials. Two grades of carbon from the Pure Carbon Co., FC-13 and 4052, were found superior to the other commercially available carbons.

During this contract period the investigations on cathode processing and performance were confined to these two types of carbon. Approximately forty cathode studies were conducted in the laboratory. Work was initiated on cathodes measuring 3 inches by 1-1/2 inches by 1/4 inches. FC-13 carbon material in each instance performed better than 4052 carbon between 2.5 volts and 3.2 volts, which is the voltage range of interest in this program. The difference in capacity between the 2 grades of carbon is illustrated in Figure 20. This is a typical discharge voltage profile of these two carbon materials at a constant current of 3 amps. With these particular cathodes the capacity between 3.3 volts and 2.5 volts is 1.8 amp hour per cubic inch (4.8 watt hours per cubic inch) for the FC 13 and only 1.2 amp hours per cubic inch (3.4 watt hours per cubic inch) for the 4052 material. The capacity from 3.3 to 1 volt is 2.9 and 2.7 amp hours per cubic inch for the FC-13 and 4052 carbon respectively. Figure 20 also suggests that after discharge, the initial charge rate for the FC-13 material is faster than the 4052 charge rate. Subsequent FC-13 carbon cathodes not only performed better than the 4052 carbon but achieved the rated capacity of 5 watt hours/in³ of cathode. In order to achieve high packing efficiencies in batteries it is desirable to increase the thickness of cathodes in order to minimize the number of cells needed in a battery. An investigation was undertaken to develop a process for producing 5 watt hours/in³ from cathodes with thicknesses greater than 1/4". Since the cathodes used in the pilot line are fabricated by gluing two pieces of carbon electrode together, this method of attack was tried. From previous work, 3/8" thick FC-13 carbon stock was available for study. Materials of this type could then be used to produce 3/4" thick cathodes. The 3/8" thick stock material of FC-13 carbon continually failed to reach the necessary capacity levels in spite of numerous modifications in the TeCl₄ impregnation method. This failure led to an investigation of the "wetting" efficiency of FC-13 carbon cathodes of varying thicknesses. During this investigation the 3/8" thick FC-13 material became suspect. For these experiments a series of FC-13 carbons of varying thicknesses (1/8, 1/4, 3/8, and 1/2") were heat treated but not impregnated with TeCl₄. This eliminated any dependence of the capacity on the impregnation procedure of the TeCl₄. The 1/8" and 1/2" thick cathodes were prepared from 1/4" thick stock. The capacity of the cathodes 1/8, 1/4 and 1/2 thick cathodes agreed to within + of - 4% while the 3/8" stock material had a capacity of 13% lower than the other three cathodes. In an effort to detect any difference in FC-13 carbon, the density of this material from various sources and of varying sizes was measured. Additional FC-13 1/4" thick stock material was

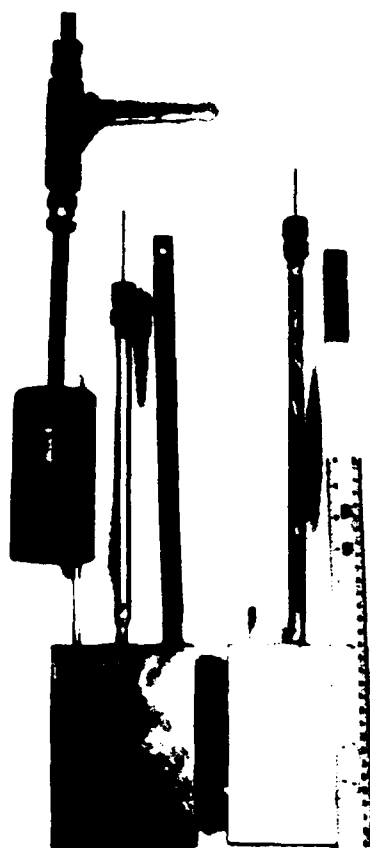
Assembled
Fitting

Spacers

Figure 17 - Cold Zone Seal and Can Spacers

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Sealed Cell
Prior to
Final Cell
Conditioning



Completed
Sealed Cell

12" Ruler

Figure 18 - Sealed Cells

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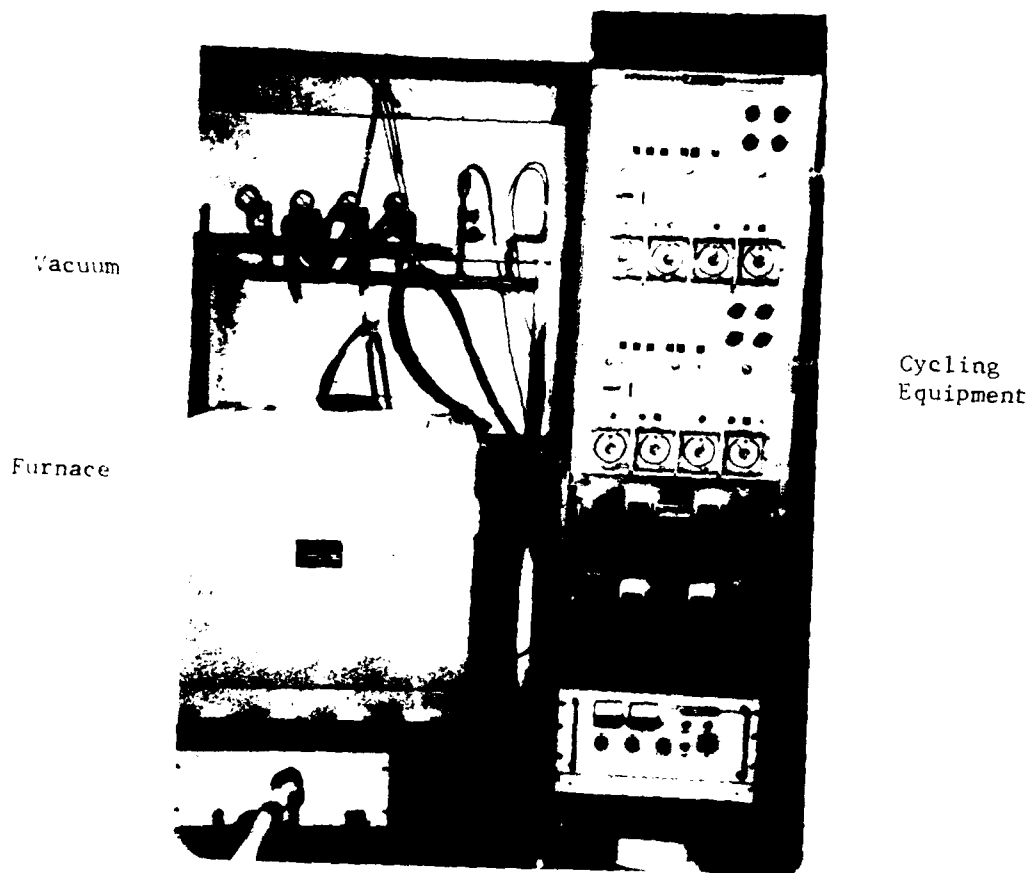


Figure 19 - Cell Conditioning Station

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CHARGE/DISCHARGE CURVES FOR FC-13 &
4052 CARBONS

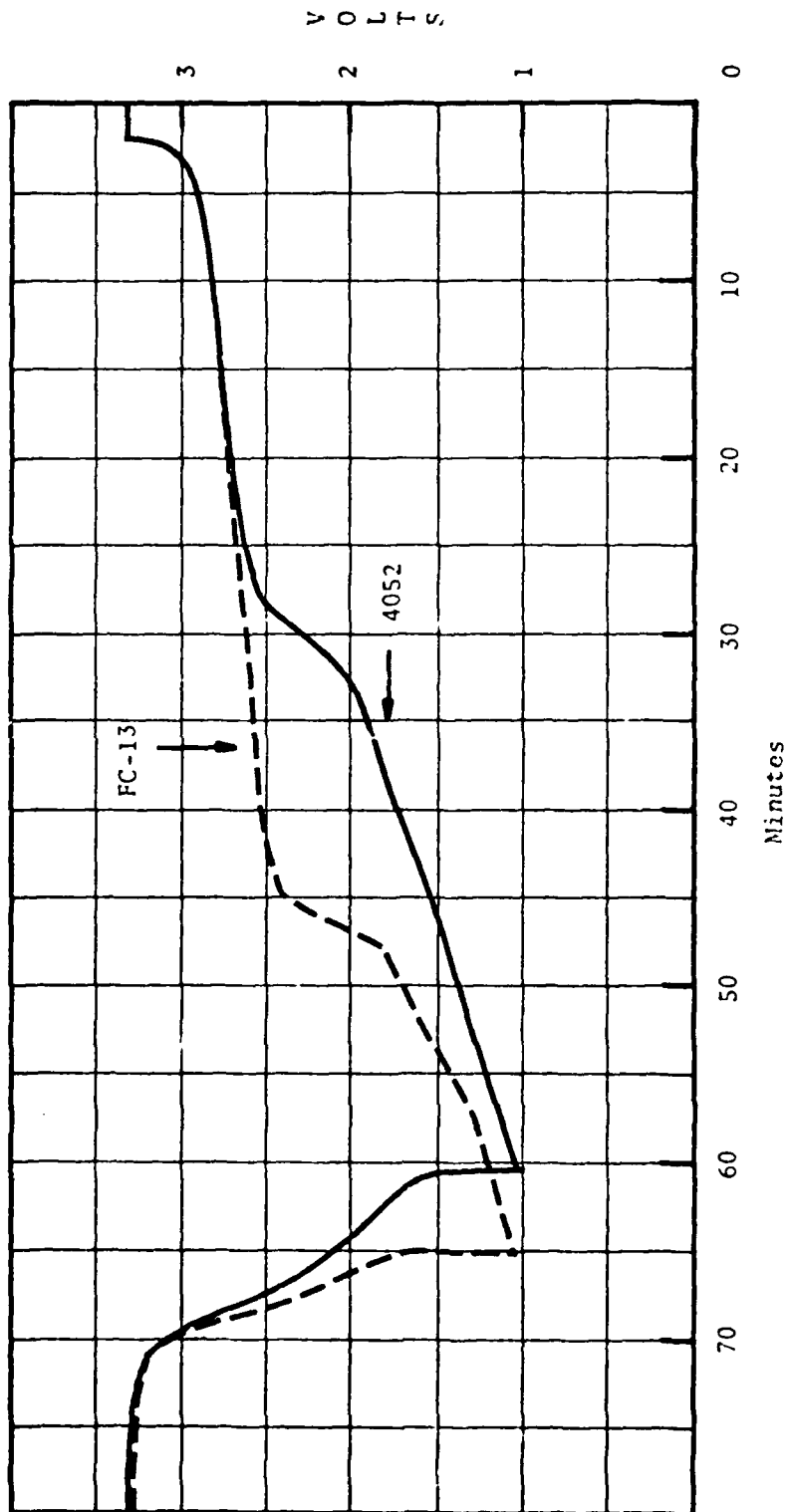


Figure 20

obtained from the ESB, Inc. Yardley Tech Center. This material and 1/4" material on hand consistently gave a density of over 0.9 g/cc. The FC-13 3/8" thick stock material had a density of approximately 0.8 g/cc. Pure Carbon Company Technical Bulletin #964 gives an average of 0.90 g/cc for FC-13.

Because of the difficulty in obtaining the rated capacity with the 3/8" thick FC-13 material, a sample of 11/16" thick FC-13 was obtained from the Pure Carbon Company. This carbon material was treated with TeCl_4 . An initial capacity of 5.2 watt hr/in was attained before decreasing to 4.6 watt hr/in. This was the first time a TeCl_4 impregnated cathode thicker than 1/4" had achieved the desired rated capacity. The failure of this cathode to maintain the rated capacity is partly attributable to two causes, (a) Solidification of the electrolyte during shutdown and subsequent stress cracking; (b) Insufficient cathode conditioning: A second FC-13 11/16" thick cathode also yielded 5 watt hr/in using a different TeCl_4 impregnation method. This procedure did not eliminate the general cracking problem and the capacity of this cathode also decreased with cycling. The cathode conditioning process is currently being revised in order to eliminate this problem in the future.

A "PACCO" grade carbon was investigated during the process improvement studies in order to compare results with the pilot line. These studies were done on cathodes of 2 in³ and compared with results obtained on cathodes of 5 in³ in the pilot line work. Results indicated good agreement between the two groups. (1.20 amphr/in³ vs. 1.25 amphr/in³)

TeCl_4 Cathode Impregnation

The procedure used for TeCl_4 treating carbon cathodes in prior work did not give consistent results. An investigation was undertaken to improve this process. In the procedure used prior to this contract the vacuum baked (850-900°C) carbon cathodes were placed into a reaction chamber in an inert environment. Powdered TeCl_4 (approximately 5 grams/in³) was placed in the reaction chamber. The chamber was then sealed and heated to above the TeCl_4 vaporization temperature allowing the vapor to permeate throughout the carbon cathodes. Several modifications to the TeCl_4 impregnation process have been studied. Three procedures have resulted in cathodes with capacities of 5 watt hour/in³. These three procedures were as follows:

- (A) TeCl_4 powder was placed in the chamber with the cathode. The impregnation chamber was then evacuated and sealed prior to heating to 450°C for 4 hours. The system was allowed to cool and the cathode was removed for electrochemical cycling.
- (B) TeCl_4 powder was placed in the chamber with the cathodes, the chamber was then evacuated and the TeCl_4 was vaporized by heating. At the maximum vapor pressure, as indicated by a pressure gauge on the chamber, hot argon was injected into the chamber. The system was allowed to cool under this positive pressure before the cathode was removed for electrochemical cycling.

- (C) The TeCl_4 powder was put into the chamber. The cathode, which had a hole going its entire length and through its center was, suspended on a stainless steel tube which was hung from the impregnation chamber lid. The tube had small holes drilled along its entire length. The system was then evacuated, heat applied, and the powder was allowed to vaporize. As the powder vaporized, a vacuum was pulled through the cathodes via the tube. The vacuum and heating was allowed to continue until the vapor pressure of the TeCl_4 started to decrease, at that point the vacuum was shut off and the system was pressurized with argon. The cathode was then ready for cycling.

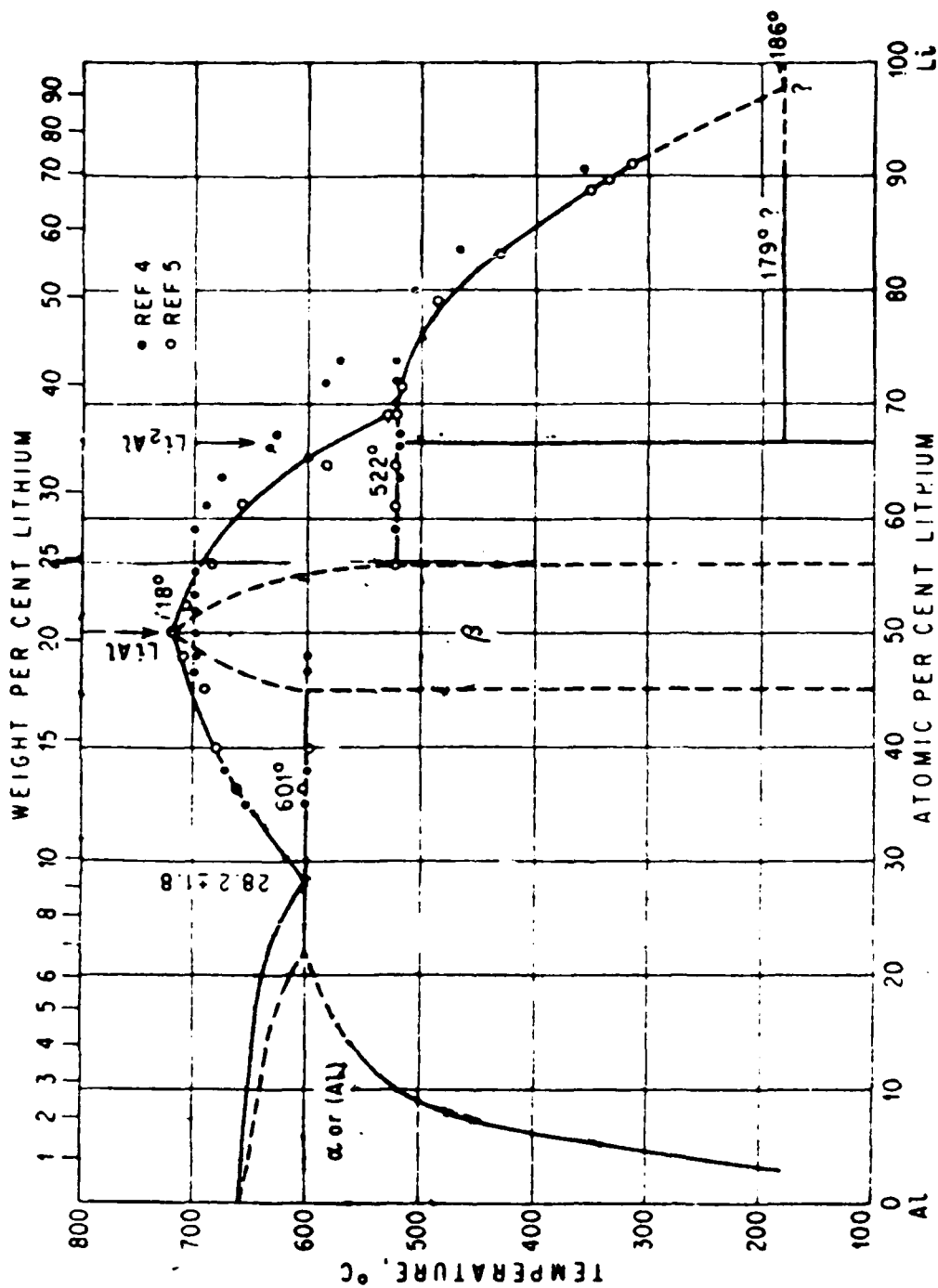
Anodes

The lithium-aluminum anodes currently used in the battery cells have been purchased from the Foote Mineral Company. The quality of the anodes has been a point of concern. The contamination of the alloys with the oxygen and nitrogen during the melting and casting operations in the normal processing environment must be minimized. However, equipment suitable for operating in an argon atmosphere cannot be presently justified because of the low volume demand for the alloy. Two alternate methods of anode production have been examined during the course of this work. They were powdered metallurgy and the electrochemical "pump-up" of aluminum. A phase diagram of the lithium-aluminum system is shown in Figure 21.

If the pressing and sintering of lithium and aluminum powder is to be successful, one phase or component must be liquified to a small degree under increases in pressure and/or temperature to cause bonding. At the same time, contamination from oxygen and nitrogen must be eliminated. If successful, the sintered anode has the following advantages: (a) provides an increase in effective surface area, (b) increases the rate of lithium migration per unit volume of aluminum, (c) reduces the possibility of forming liquid lithium on the surface of the anode, (d) reduces the effective anode current density, (e) permits compositions of lithium and aluminum to be formed as required, and (f) presents the opportunity to assemble cells in an essentially full state of charge.

An effort was made to form several pellets from powdered aluminum and lithium in the dry box. The pressing of an 18 wt.% Li-Al mixture resulted in a few very bright, shiny 1/2" diameter pellets ranging from about 1/16" thick to about 3/8" thick. Reasonable "green" strength was evident. Attempts to improve the pellet strength by heating at about 150°C always resulted in the disintegration of the pellets. This latter difficulty was thought to have been the result of an ever increasing oxygen level condition that was unknown at the time of the experiments.

The mixed powders of lithium and aluminum indicated the formation of the beta phase material when pressed at room temperature with or without subsequent heating. Beta phase material, as evidenced by previous anode investigations expanded and became porous. The same effect has been shown repeatedly in each of the experiments. The pellets became darker



Li-Al PHASE DIAGRAM

Figure 21

in color, dull grey to black. They lacked any tendency to bond to adjacent particles. These effects were due to a phase change rather than to dry-box contamination. Further experiments were conducted to press pellets, and to heat said pellets subsequently in the die. Equipment limitations made it necessary to place weights on the die plunger rather than taking advantage of the press pressure. As the beta phase formed, it extruded the sample out of the bottom of the die. A clamping arrangement was used subsequently to prevent the extrusions but bonding was not accomplished. Consideration was given to reducing the percent lithium to 12 wt.% to avoid 100 wt.% beta phase formation and to attain a measure of bonding capability. A lack of adhesion between the 12 wt.% particles was also noted.

All experiments had been conducted in the dry box atmosphere using a Paar Pellet Press capable of exerting a maximum pressure of 2000 p.s.i. Attempts to keep the pressure applied while heating, or to heat while pressing, were literally impossible using this equipment. Insufficient applied pressure during heating, and the concurrent phase formation usually resulted in cracked pellets.

It was decided that a few liberties must be taken to prove the procedure. An 1-1/2" diameter die was charged with the anode mixture while in the dry box. The die plunger was inserted as far as possible by hand. The die assembly containing the charge was removed from the dry box and immediately placed in a Pasadena, heated platten, hydraulic press. A pressure of about 2000 p.s.i. was applied while the heat was allowed to build up on the die. No additional pressure was applied, but a pressure from formation of the beta phase Li-Al was noted. The pressure gradually increased to a maximum of 18,000 p.s.i. The high value of 18,000 was unexpected.

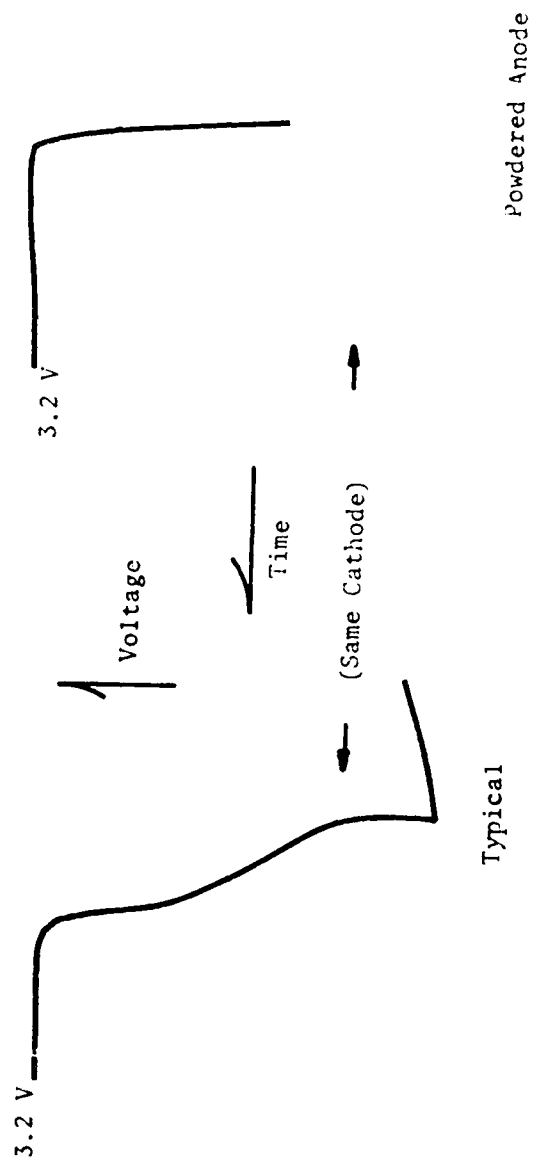
Four pellets were made by the above procedure, (a) 19.74% Li, (b) 20.00% Li, (c) 12.00% Li., and (d) 12.00% Li. The pellets were about 1/8" thick, about 5 grams each, and 1-1/2" in diameter.

Each pellet was pressed from the die after the die had been cooled to room temperature. The pellet was then quickly returned to the dry box. The pellets were examined each day to note any color changes, cracks, or breaks. Only the 12 wt.% discs had not changed after eight days.

It appears at this time that compositions can be fabricated up to about 14 wt.% lithium.

Two 1-1/2" diameter x 1/8" thick discs of 12 wt.% lithium/aluminum anodes made from powder were placed in an open cell and tested. Prior to testing, a normally functioning cathode was run. The same cathode was then used immediately for the anode test. Two charge/discharge cycles were run. Interesting results were obtained. First, a change in the shape of the charge curve was noted. See Figure 22 and Table 1. The typical curve shows two inflection points while the curve of the powdered anode shows none. The total charge time is less than half the typical time. A sudden drop in potential during the second discharge cycle indicated

COMPARISON OF CHARGE CURVES



Constant Voltage Charge

Figure 22

TABLE 1
POWDERED ANODE CYCLE DATA

<u>Function</u>	<u>Voltage Range</u>	<u>Total Time</u>
Charge	1.80 to 3.30	1.5 min.
Charge	3.30 to 3.32	95.0 min.
Discharge	3.32 to 2.50	55.5 min.
Discharge	2.50 to 1.82	9.5 min.
Charge	1.82 to 3.30	0.6 min.
Charge	3.30 to 3.32	19.5 min.
Discharge	3.32 to 2.8	7.5 min.
- Oscillations then failure -		5.0 min.

malfunction. The test was terminated and the cell was examined. Only 1/4" diameter section of the anode was intact and the other anode was broken into several large sections. The anode discs being only 1-1/2" in diameter were not held securely by the anode screens as in the normal case but the large loss of anode stock on the one side of the cell has not been explained. The loss may have been due to beta phase Li-Al small particle formation and subsequent fall-off and dropping to the bottom of the cell. The results indicate insufficient strength and lack of bonding between particles, but an ability to improve charge rates.

The second method (electrochemical "pump-up") involves the electrochemical transport and reduction of lithium ions from the eutectic melt to the aluminum sheet. Here lithium metal is combined with the aluminum to form the alloy. Two sheets of capacitor grade aluminum were simultaneously charged at a rate sufficient to obtain anodes of 18% (weight) lithium assuming 100% current efficiency. After completion of the experiment, the samples were sent to the analytical department for analysis. The two plates contained 13 and 20 wt.% lithium. The 20 wt.% anode could not be removed for analysis without breaking. The electrochemical formation of thin Li-Al anodes up to approximately 20 wt.% Li appears to be possible. The advantages of electrochemical formation of anodes include, (a) elimination of mechanical fabrication, (b) the reduction of costs, and (c) the minimizing of cell contamination with oxide and nitride impurities.

Separators

The problem of cost reduction in the materials and fabrication of the separators was considered. In order to minimize the boron nitride fiber waste in the production of separators, process changes were studied, and possible alternate low-cost separator materials were investigated.

Hercules, Inc., has supplied a high temperature polyamide resin material which they believed would be suitable for incorporation into a cell as a separator. Unfortunately this material as treated by the vendor is very fragile and appears to decompose in the molten salt electrochemical environment. The decomposition is the result of the high temperature (500°C) of the cell and/or the electrochemical processes occurring between anode and cathode. Further tests using differently treated samples are warranted before ruling out this material as a separator. The considerable difference in cost, \$32.00 per pound for the Hercules material versus \$600 per pound of boron nitride fiber, is a further incentive to continue experimentation on this material. Other alternatives to boron nitride fiber are also being considered.

A process for in situ fabrication of boron nitrile separators is being investigated. The technique involves pulling the boron nitride slurry directly through a stainless steel screen thereby forming the separator in place on the screen. Such a fabrication method would save 20% in boron nitride fiber and save considerable time and cost in production. Pilot line quantity and quality testing of boron nitride separators formed in this manner are necessary before a final evaluation of this technique can be made.

High Temperature Cements

An investigation was initiated to either find higher conductivity graphite cements or to increase the conductivity of cements commercially available. These cements are used in the fabrication of pilot line cathodes and to attach the graphite current collector to the carbon cathodes.

An attempt was made to increase the conductivity of cement by the introduction of tungsten metal powder. Unfortunately with the two cements tested thus far, Union Carbide's C-9 and Atlas' Furathane, the conductivity decreased upon the addition of Tungsten powder. Cements are being obtained from various manufacturers with the object of low cost, high conductivity, and high holding strength.

Seal

The hot zone seal has been found to be a weak point in the present cell designs. Therefore, in the present cell studies a cold zone seal has been used to allow work to proceed on other cell components while parallel studies are developed on the best location for a seal as well as the best means of construction. The seal area of the cell by design is usually situated on the topside of a cell. In this position it is subjected to a liquid-gas interface and a high cell potential which can promote corrosion and subject it to the collection of floating corrosion products from other cell components. It appears that a design in which the seal is on the side or bottom, which would provide a constant submersion in the liquid, would be superior. In this position the liquid would promote washing of the seal area and suppress chlorine evolution and corrosion when the full charge state of the cell is approached. Actual laboratory study on seals in working cells will be undertaken when the ESB, Inc. personnel have established the quality control on the other cell components.

Formation Tank Design

In the present formation tank the largest cathode that can be processed is about 7" x 7" x 5/8". A look at what size cell might be needed in the final prototype battery for a fork lift truck indicated a much larger cathode would be needed than could be processed in the existing tank which is 22" x 38" x 12".

A critical examination of the present salt tank design resulted in modifications that retain the former length and width but adds about 20% to the depth and the rearrangement of fixtures to increase the usable interior space. A larger dump tank will not be required. It will now be possible to make cathodes from 4" x 4" to 10" x 12" and from 5/16" thick to 1.1" thick and to increase the number of cathodes from 6 to 12. The detail drawings of this tank, lids and associated hardware are completed and are ready for submission to vendors for bids. Discussions have been held with Vacuum Atmosphere Corp. on the design of the inert atmosphere box in which the tank will be installed. The width of the dry box will

be increased by six inches to facilitate the movement of lids. The top portion of the box will be raised to permit the higher mounting of the hoist used to lift the lids from the tank. This will enable the moving of a lid with cathodes over another lid still on the other half of the tank. It is anticipated that the new formation tank and inert atmosphere box will be purchased during the next contract period.

Packaging Studies for the Forklift Truck Application

A subcontract was issued to Exide Power Systems Division of ESB, Inc. to provide guidance on the future design of a battery for a forklift truck application. This design was not intended to be a firm detailed design but rather one to check the feasibility of packaging a battery to fit in an existing forklift truck, determining the size cells that might be used, and determining the watt hour per lb. and watt hour per cubic ft. that might be expected upon the successful completion of a molten salt battery. These studies are given in detail in Appendix I.

DISCUSSION

Dry Box System

The dry box system has been completely reassembled (Figures 9, 23, 24, 25). This system provides a work area free of moisture, oxygen, and nitrogen where materials such as lithium-aluminum alloy, lithium chloride, potassium chloride etc. can be handled.

This dry box system is a completely closed recirculative system. Additional argon gas is added as required to replace losses due to pressure control or in refilling an evacuated ante-chamber. During normal operations the recirculating gases pass through exterior dry trains and getter furnaces before returning to the dry box system.

Certain steps are followed to attain the initial inert atmosphere. The system is repeatedly purged and diluted with fresh argon gas until a relatively low moisture content is indicated on the monitor. Then the atmosphere is circulated through the dry trains and the unheated getter furnaces until the dew point is -40°C or lower. Then the getter furnaces are turned on to remove oxygen and nitrogen contamination. When it has been determined that the oxygen concentration is 1 ppm or less, the system is ready for use. The constant operation of the dry trains and getter furnaces is necessary to remove air contamination that diffuses through the glove material and from materials introduced into the dry boxes during normal operations.

To maintain an atmosphere purity level of less than 1 ppm oxygen, moisture and nitrogen, the system must not only be completely free of leaks initially but maintained leak-free on a day-to-day basis. The system includes every joint, fitting, and connection from the reserve gas connections throughout the entire system and most especially the finger tips of the gloves.

Operation of Dry Trains and Dry Boxes

Fully evacuable ante-chambers, Figure 25, are provided as an airlock so that materials, parts, and tools may be passed in and out of the work area without contamination to the internal atmosphere.

Both automatic and manual pressure controls have been provided to maintain the gloves at a workable pressure as well as preventing the rupture of gloves and the breakage of windows.

Molten Salt System

The molten salt system was reconstructed and operated for seven weeks before a failure occurred. Approximately 200 pounds of molten salt were stored in a dump tank and through vacuum and pressure controls could be circulated into the formation tank for cathode processing.

A leak developed in the dump tank and caused failures in the associated heaters on the tank and piping. The leak was probably a failure due to

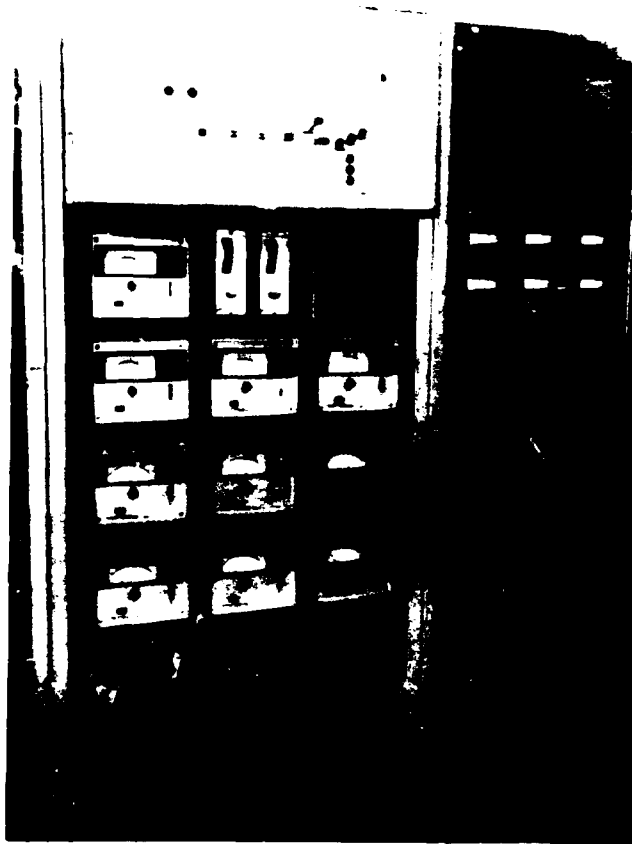


Figure 23 - Control Panel for Molten Salt System

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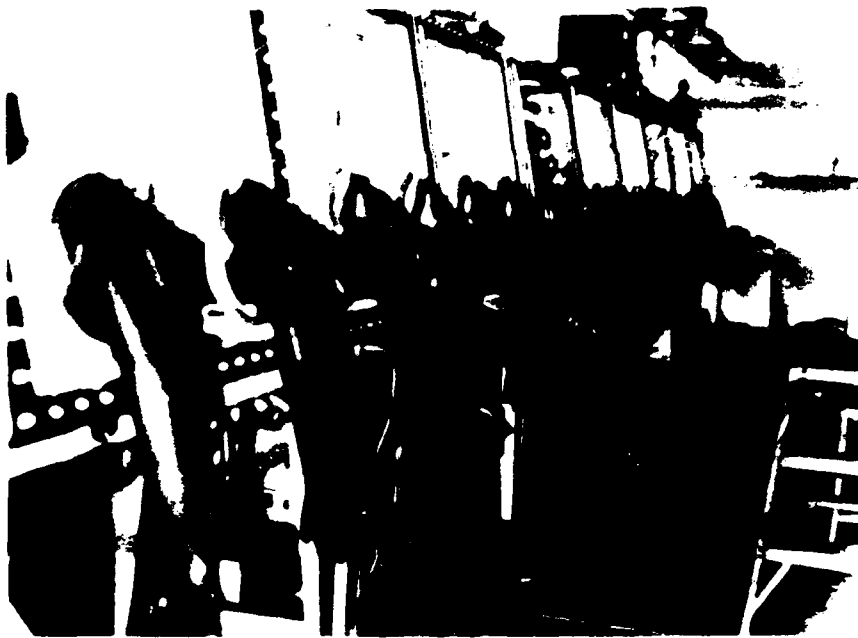


Figure 24 - Prebox Assembly and Storage Areas

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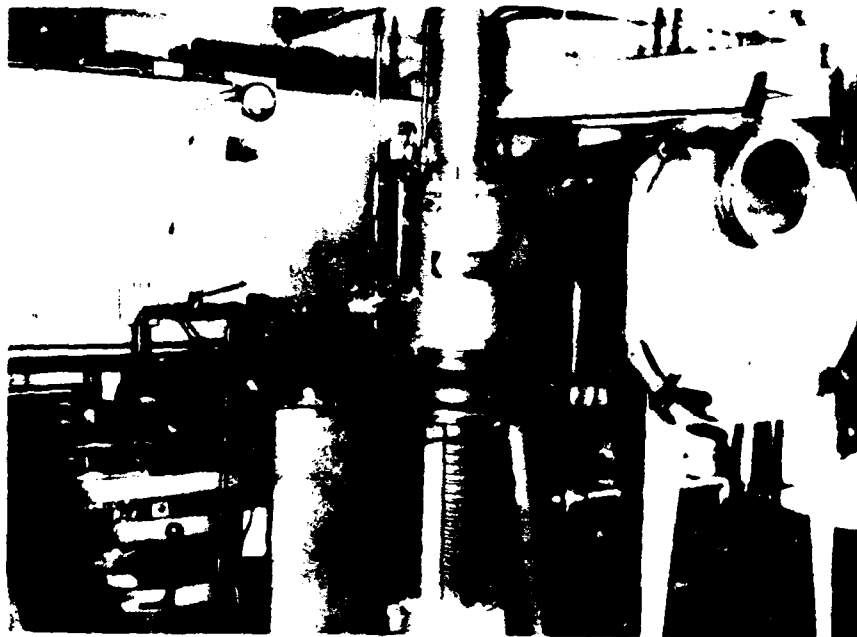


Figure 25 - Large Ante Chamber for Admitting Components Into
The Dry Box

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internal corrosion at a weld from the long period of storage of the tank (3 years) with salt present in the pores of the metal. A new tank has been ordered and will be delivered by November 15.

The pilot line is still in operation, but the formation tank must be emptied and refilled by hand.

Cell Production

Eighteen cathodes were processed in the formation tank during the course of this work. Most of these cathodes were lost in some phase of cell production as they were being used to familiarize the ESB Inc., personnel with the Carb Tek® technology.

Two cells were constructed using 13 mil capacitor grade aluminum as anodes. The results from these cells were very favorable and significant. Typical charge-discharge curves for a cell are shown in Figures 26 and 27. These cells were cycled between 3.2 volts and 2.5 volts. The lithium content of the anode was cycling between 11 wt.% and 4 wt.%. These were the first cells of the compact configuration (Figure 6) with TeCl_4 containing cathodes that were successfully cycled with an aluminum anode. Successfully cycling the anode to the 4 wt.% lithium level indicates that it may be possible to use less anode material in cells than was previously thought.

Both cells were run to failure (believed to be mechanical in nature). The best cell ran for 146 cycles.

Anode and Cathode Processing Improvement

Work with aluminum sheet and powders of lithium and aluminum have been shown to be two feasible alternates to cast lithium-aluminum anodes. This work indicates that reduction in cell contamination levels as well as cost reductions can be expected in the near future.

TeCl_4 treating processes have been developed which demonstrate 5 watts hours/in² of cathode. This capacity has been demonstrated on cathodes up to 11/16 of an inch thick. If the transfer of these processes is successful to the pilot line operation, cells capable of delivering at least 25 WH/lb will be possible.

Preliminary Battery Design

The final report on the preliminary battery design by Exide Power Systems of ESB, Inc. is included in Appendix I. This study shows that a Carb Tek® battery can be designed to fit in an existing fork lift truck. However, some configurations of cells cannot be used because of dimension restrictions of the compartment (as opposed to volume restrictions). With these restrictions, allowing for insulation and inter cell connections, batteries can be designed that would deliver approximately 20 watt hours/lb.

TYPICAL CHARGE CURVE
CELL #106 PUMP-UP ALUMINUM

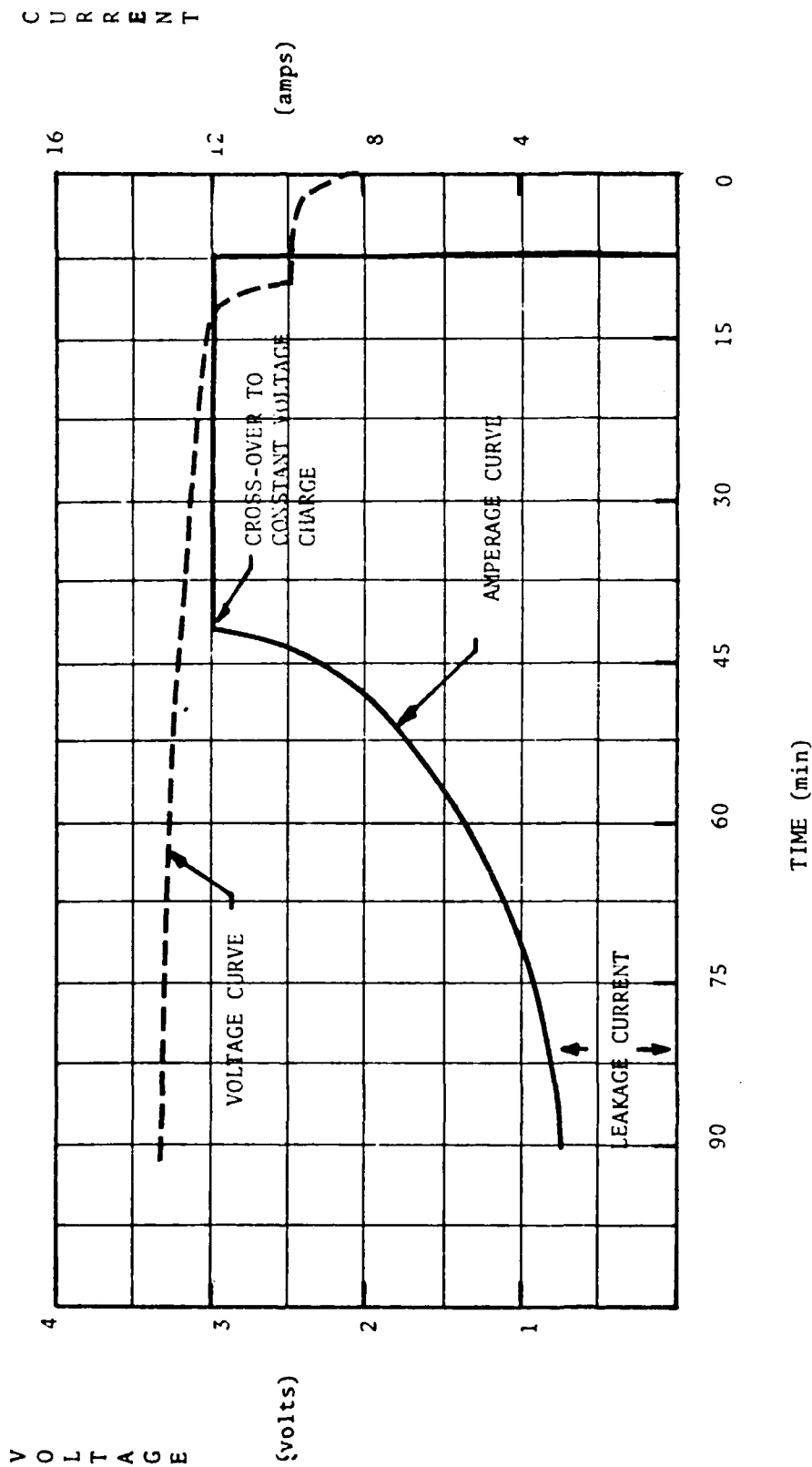


Figure 26

TYPICAL DISCHARGE CURVE
CELL #106 PUMP-UP ALUMINUM

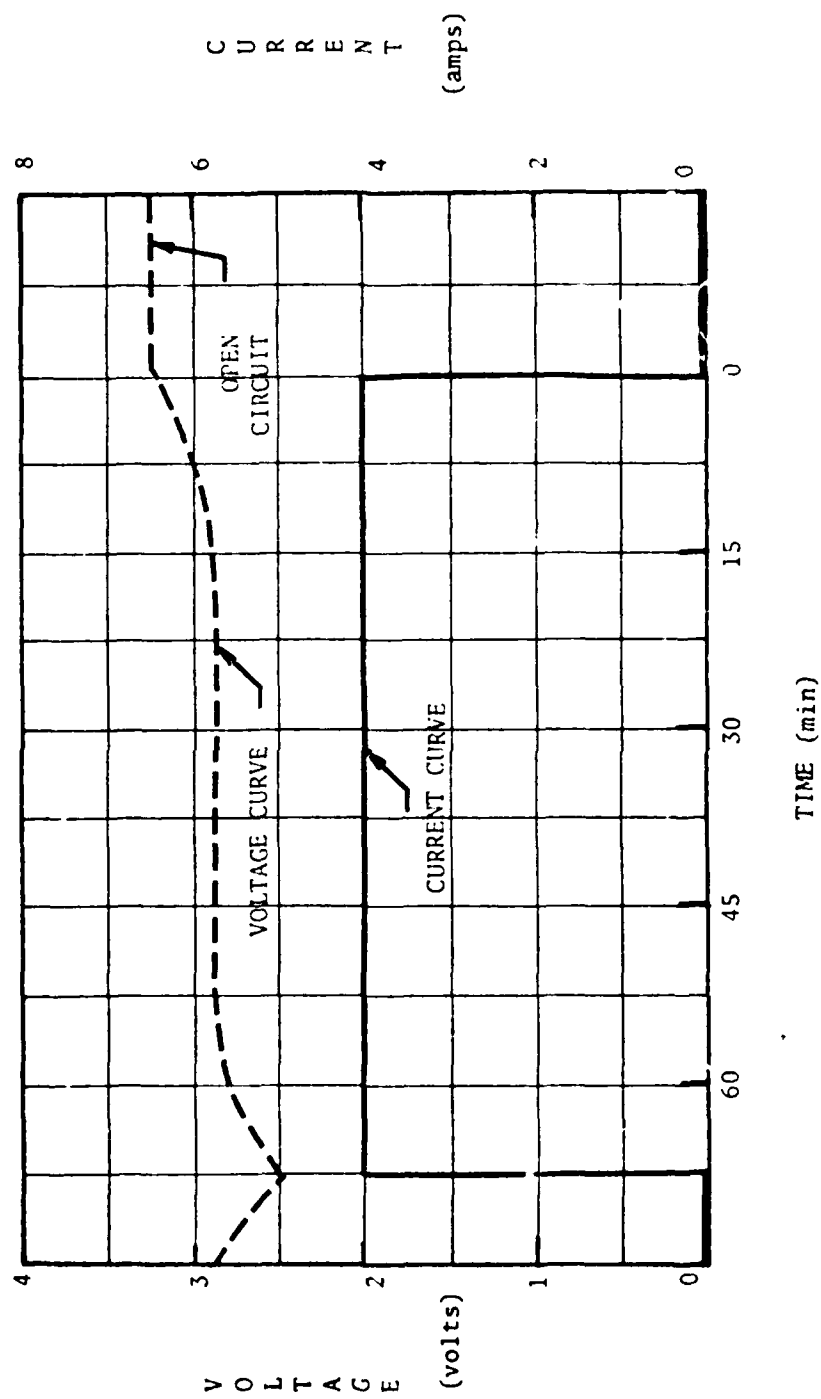


Figure 27

Cell Testing

Testing of battery cells will be carried out at the Yardley Tech Center of ESB, Inc. A testing procedure (Appendix II) is being readied for testing of cells produced under future contracts.

Quality Control

Quality control represents the key to producing good reproducible cells. A quality control program must be set up to first, keep the dry box inert atmosphere at the needed quality level; second, to keep the quality of materials and cell construction at a constant high level; and third, to monitor, record, evaluate, and determine any measurable factor affecting the quality of the cell or procedures in construction of the cells.

The major work performed in this area this past year has emphasized the dry box inert atmosphere. Work has not only concentrated upon monitoring the atmosphere but also setting up a maintenance plan to collect data and establish a preventive maintenance program.

Samples of salt, anodes, cathodes, and other parts of cells being constructed in the pilot line have been sent out for oxygen analysis. Oxygen in the ppm range for such samples must be analyzed by neutron activation. It is believed that knowing the oxygen level of the components of the complete cell and decreasing these levels will result in more reproducible and better quality cells.

The final area in evaluating cells is the "Post Mortem." The cell is dissected in an effort to determine reasons for cell failure.

Arrangements have been made through Ft. Belvoir to have Naval Research Laboratory (NRL) Personnel discuss results of their "Post Mortem" investigations regarding cells previously delivered to Fort Belvoir.

Cells produced on the pilot line have been examined on two occasions after failure. No obvious fault was found in one, while the other cell shows an electrical short after 146 cycles. A request will be made to have NRL examine the unit.

Seal

The seal on a molten salt battery must be a hermetic seal. That is, there must be no loss of electrolyte or internal gas pressure and there must be no entry from the outside environment of any air due to pressure or concentration gradients.

The selection of materials and fabrication techniques for seals had been an active area of research and development in Sohio prior to 1971. Eutectic oxide seals, fluoride salt seals and brazed seals were initially

tried. A system using a tungsten rod, boron nitride, lava, and 303 stainless steel was arrived at from thermodynamic studies. These materials were combined into a mechanical compression seal. This seal is described in U.S. Patent 3,472,701. The two drawbacks of this seal were (a) too high a gas leak rate (110 mgr. O_2 /yr @ 500°C) and (b) it was too bulky in volume and weight. From this compression seal evolved the swaged seal. Swaged seals have been produced with leak rates as low as 3 mgr. O_2 /yr at 500°C. A good description of swaging is given by Strohecker (2)² and ASM (3). An example of a swaged product is a sheathed thermocouple. In previous work the variable purity, strength and machinability of the commercial hot pressed boron nitride have caused considerable variation in seal performance.

In 1969, a contract was awarded to Battelle-Columbus to study the swaged seal. This study was broken down into five task areas. They were the following: (1) Selection of sheath materials, (2) fabrication of test seal assemblies, (3) experimental evaluation of fabricated test seals, (4) analysis of results, (5) "post mortem" examination of Sohio cycled cell seals. From the results of this Battelle study, prior chemical research, literature and thermodynamic considerations, an outline of tasks for improving the production of swaged seals has been assembled as follows:

- (1) Sheath selection should zero in on the lowest thermal expansion metals compatible with swaging, welding and cost. Possible materials are the Fe-Ni Invar type series or Fe-Co-Ni Kovar alloy.
- (2) Prior to swaging, high purity boron nitride should be hydropressed onto the tungsten rod. This will insure the lowest contamination and the maximum density.
- (3) All machining on the tungsten rod should be done prior to seal assembly. This will eliminate the vibration stresses set up in the seal.
- (4) The swaging operation should be optimized to get the maximum boron nitride density and interface bonding. Examples of possible swaging modifications are: (a) thermally cycle the cold swaged seal to 500°C followed by a single final swaging pass at room temperature, (b) swage entirely at elevated temperatures to the final dimensions, (c) the use of magnetic forming (4) to achieve the desired compaction and swaging.
- (5) The tungsten rod should be coated with a protective layer of stable ceramic to minimize corrosion by oxygen or chlorine. The most promising material is silicon carbide.
- (6) A gas tight seal at the air interface of the seal should be made. Commercial sealing glasses such as those listed in Table 2 are possibilities. The selection of glasses and the design of the seal should be done in conjunction with a glass supplier.

- (7) Quality control procedure should be developed for all materials and fabrication steps. Useful techniques for evaluating finished seals are helium leak checking, X-ray radiography and neutron radiography.

Implementing these tasks should be done in conjunction with Battelle-Columbus. They have readily available most of the needed equipment and possess expertise in materials and fabrication techniques.

Future Manufacturing Considerations

One large and obvious problem is the set-up, operation, and maintenance of a production assembly line in an inert atmosphere. To obtain a background in this area a visitation was made to Universal-Cyclops Speciality Steel Division in Bridgeville, Penna. In 1956 they installed an inert atmosphere metal fabrication facility for the working and forming of metals such as W, Ti, Mo, Cb, Ta, and their alloys. This facility measured 97 feet long x 42 feet wide x 23 feet high. Presently this facility is not in operation. Workmen entered the facility in spacesuit type suits. Much of the actual work could be done through remote controls from the exterior of the facility. During fifteen years of operation their argon losses were 10-15% per day. This is not too bad considering the volume of the facility and that helium leak detectors were not available when this facility was constructed. Presently, it appears that a combination of the good features of this facility and our present pilot facility should yield a very workable production facility.

It is anticipated that during the next phase of this work a conceptual plant layout will be started and, where practical, testing of components such as conveyors will be evaluated.

TABLE 2

Glass Sealing Material Properties

Hard Glass	Vendor	CTE (RT-500°C) μin/in/°C	Strain Pt. °C	Seal Temp. °C	ReHeat Temp. °C	ΔExp. Mismatch PPM	Vol Resist (@ T (ohm Cm))
7056	C	5.7	470			130 (Kovar)	10 ^{8.3} @350°C
7720 (None x)	C	4.3	485			120 (W) 860 (Kovar)	10 ^{7.2} @350°C 10 ^{5.8} @500°C
<u>Solder Glass</u>							
00130 (SG-7) (Vitreous)	O-I	6.1	443	615 45 min.	550 bake	For (Kovar)	10 ^{10.5} @350°C
00331 (EN-1) (Vitreous)	"	6.7	431	740 15 min.	700 bake	"	10 ^{7.2} @350°C
7574 (Pyrocera No. 45) (Devitrifying)	C	4		750 60 min.	700 bake 650 contin.	For (W)	10 ^{11.6} @350°C
QJ-231 (Vitreous)	P	4.6		790			
QJ-235 (Vitreous)	"	4.0		690			
QJ-236 (Vitreous)	"	6.0		560			

Vendors: C - Corning
O-I - Owens Illinois
P - Pemco Div. SCM

CONCLUSIONS

The inert atmosphere pilot line facility has been completely refurbished and is in excellent working order. An argon atmosphere with less than 1 ppm oxygen, nitrogen, and water can be maintained.

The replacement of the dump tank will provide an operable salt handling system.

Process improvement work and limited pilot line work indicates cells capable of delivering 25 watt hours/lb. will be achievable.

Battery design concepts indicate a finished battery with large, thick cathodes could deliver as much as 30 watt hours/lb. ($\sim 3500 \text{ WH/ft}^3$).

The evolution of a battery company (ESB, Inc.) has been very successful. The ESB personnel are now very knowledgeable in the Carb Tek® battery technology. They will now be able to apply battery production and engineering methods to further the development of the Carb Tek® battery.

RECOMMENDATIONS

Work should continue in the direction of building reproducible cells while incorporating the improvements found in the process development work. After establishing reproducibility, the cell size should be increased to utilize cathodes of the largest size possible in the present formation tank (7" x 7").

A program for seal work should be developed with Battelle-Columbus.

Battery design work should be expanded to include studies on electrical connections and more definite heat balance studies.

A new formation tank should be purchased and installed. This tank should be capable of handling the cathode sizes described in the preliminary battery design work.

Quality control standards should be developed for all cell components and procedures.

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2. Strohecker, D. E. "Swaging" in Metal Deformation Processing Vol. III by F. W. Boulger et al, Eds.. Battelle, Columbus Labs DMIC Report 243, June 1967, p. 104-116.
3. "Rotary Swaging of Bars and Tubes," Metals Handbook, 8th Edition Vol. 4. Forming American Society for Metals, 1969, pp333-346.
4. Boulger, F.W., "High Velocity Forming Electromagnetic Forming, in Metal Deformation Processing, Vol. II, by Boulger et al eds; Battele Columbus Labs DMIC Report 226, July 1966, pp 26-27.

APPENDIX I

DEVELOPMENT OF Li/Cl_2 MOLTEN SALT BATTERY

CONTRACT DAK02-73-C-0493


U.S. ARMY MOBILIZATION EQUIPMENT RESEARCH AND
DEVELOPMENT CENTER

STANDARD OIL COMPANY (OHIO)

SUBCONTRACTOR: ESB INCORPORATED

August 1974

CONTINUATION OF STUDY LEADING TO A
PRELIMINARY DESIGN OF BATTERY FOR FORK LIFT TRUCK
EXIDE POWER SYSTEMS DIVISION


Klaus K. Berj
Project Manager EPSD

SONIO PROJECT

Subject: Preliminary Design
SONIO Carb-Tek (R) Battery
Battery for Fork Lift Truck
Contract DAAK 02-73-C-0493

Purpose: Continuation of Study of Design
Parameters of SONIO Carb-Tek (R) System leading to optimum
design of a 25 KWH power package into an existing fork
lift truck

General Considerations:

The previous report reviewed the general principles of construction and operation of the SONIO Carb-Tek (R) System and examined the feasibility of designing a 25 KWH molten salt battery package into the available space previously occupied by a lead acid battery in an existing fork lift truck presently operating at MERDC.

By direction this study examined the possibility of filling the available compartment by using cells containing 3/4" and 7/8" thickness cathodes.

Without examining in any detail the volume required to perform the interconnection and termination of individual cells, multiple cell units, or the total power package as well as the insulation envelope, it was concluded that the 8.09 cu. ft. available was sufficient to house a 25 KWH power package.

Packaging Study:

To further refine this study and in fulfillment of the task of examining the packaging considerations of this system let us analyze the available space and the cell variations possible.

The 2000 lb. capacity truck operating at MERDC has an available space of 31.5 in. x 19.625 x 22.655 = 8.09 cu. ft. The energy package required to meet the MERDC specification is 25,000 watt hours and the volumetric energy density, therefore, must be $\frac{25,000}{8.09}$ or 3090 w. hr./cu. ft.

cont'd

From records obtained from the SOHIO Laboratories the SOHIO #6 cell demonstrated an energy delivery of 5 W. Hrs./cu. in. of carbon. The carbon cathode in this cell was 4" X 4-3/8" X .330 = 5.775 cu. in. The cell dimension of this cell was .625 X 4-9/32 X 6" or 16.05 cu. in.

The volumetric energy density of this cell can readily be calculated to be

$$\frac{5.775 \times 5}{16.05} \times 1728 = 3108.8 \text{ W. Hr./cu. ft.}$$

Using similar design concepts and materials of construction, a #9 cell was developed which contained a carbon cathode of dimensions

$$7.203 \times 7.203 \times .875 = 45.397 \text{ cu. in.}$$

The external cell dimensions of this cell are $7 \frac{29}{64} \times 9 \frac{13}{64} \times 1 \frac{9}{32} = 87.76 \text{ cu.in.}$

The volumetric energy density of this cell is $\frac{45.397}{87.76} \times 5 \times 1728 = 4469.34 \text{ w.hr/cu.ft.}^3$

Using the general design parameters of the #9 cell and varying the cathode thicknesses from 5/8" to 1" is a study which was conducted to determine what optimum volumetric energy densities could be achieved. In addition to the dimensional constraints we have taken the following into consideration:

1. Assuming a nominal end voltage of 2.5 volts for this system, this package will require 12 modules for the required end voltage of 30 volts.
2. For this study, super insulation only was considered, requiring a 1" thick envelope around the entire battery. This insulation envelope, therefore, reduces the available space for cells to 29.5 in. X 17.625 in. X 20.625 in.

Analyzing the #9 cell design we find the difference between the cell can and the carbon cathode to be:

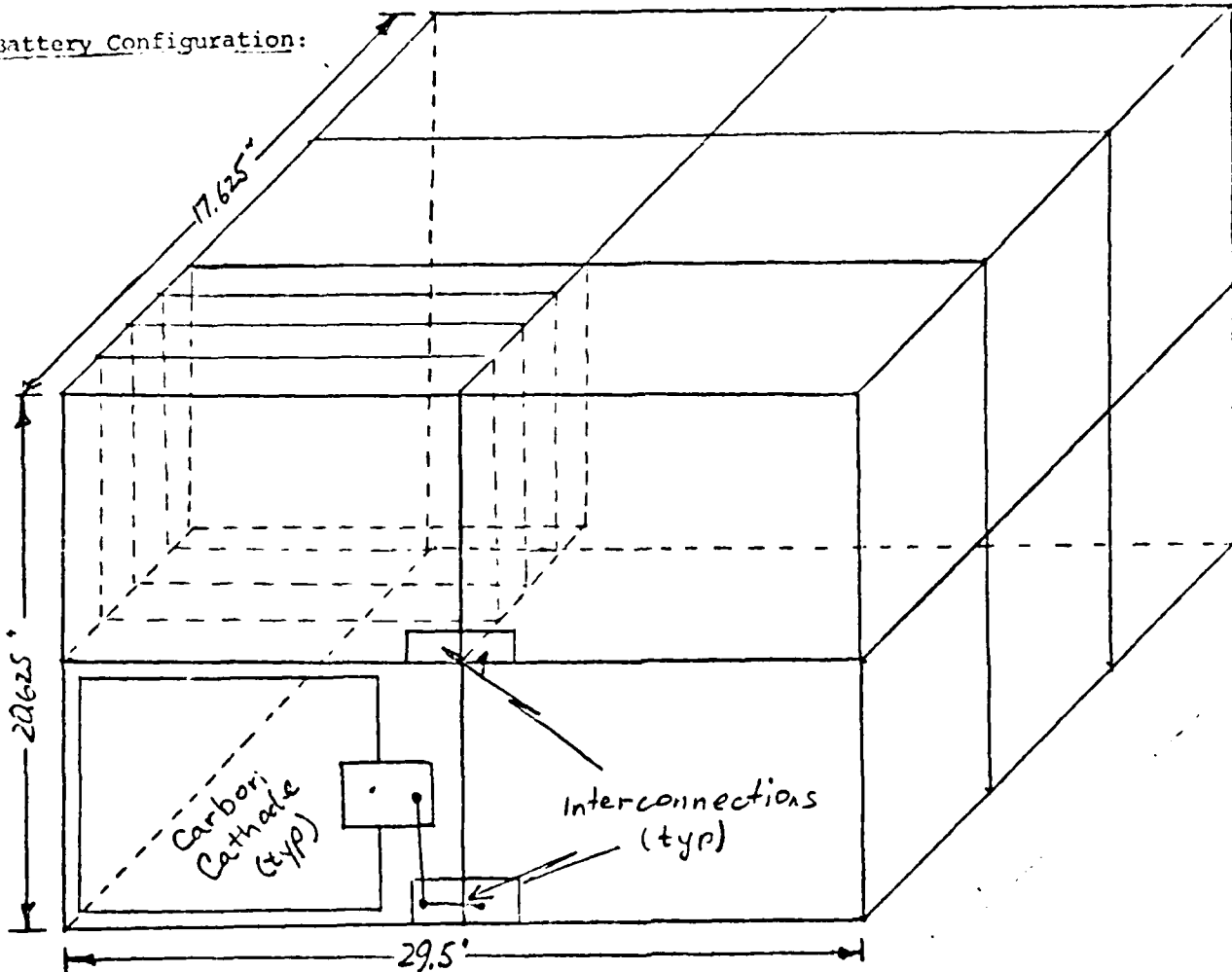
$$\text{Length} - 9 \frac{13}{64} - 7 \frac{13}{64} = 2.00 \text{ in.}$$

$$\text{Width} - 7 \frac{29}{64} - 7 \frac{13}{64} = .25 \text{ in.}$$

cont'd

Using the battery configuration and interconnection concept previously reported,
we have the following:

Battery Configuration:



In order to utilize the available space in an optimum fashion the cells are placed in series in the 17.625" dimension. The 12 modules are placed 3 across the 17.625" dimension. In order to take advantage of obtaining the largest cathode area possible the modules are placed 2 X 2 across the 20.625" X 29.5" face. Refer to diagram above.

cont'd

Maximum Cell Dimensions in this package will be:

$$\text{Length } \frac{29.5}{2} = 14.75 \text{ in.} \quad \text{Width } \frac{20.625}{2} = 10.3125$$

Maximum carbon cathode in these cans:

$$\text{Length } 14.75 - 2.00 = 12.75" \quad \text{Width } 10.3125 - .25 = 10.06$$

Therefore, the respective batteries will have the following characteristics:

<u>Carbon Thickness</u>	<u>Carbon Volume</u>	<u>Watt Hours per Cell</u>	<u>No. of Cells Required</u>	<u>Total Watt Hours</u>
5/8"	80.165	400.825	12 X 6 = 72	28,859
3/4"	96.198	480.99	12 X 5 = 60	28,859
7/8"	112.23	561.15	12 X 4 = 48	29,935
1"	128.26	641.3	12 X 4 = 48	30,782

All of these designs, however, are not feasible or optimum since the required cells can not be placed within the confines of the 17.625" dimension which is affected by the cathode thickness.

<u>Carbon Thickness</u>	<u>Cell Thickness</u>	<u>Cells Across 17.625 Dimension</u>	<u>Cells Required in 17.625 Dim.</u>
5/8"	1.03	17.11	18
3/4"	1.155	15.25	15
7/8"	1.28	13.78	12
1"	1.405	12.54	12

Revising the carbon thicknesses to three dimensions in order to take full advantage of the 17.625" dimension we obtain -

<u>No. of Cells in 17.625" Dim.</u>	<u>Cell Thickness</u>	<u>Carbon Thickness</u>
3 X 6 = 18	.979	.574
3 X 5 = 15	1.175	.77
3 X 4 = 12	1.465	1.063

cont'd

Using these carbon cathode thicknesses the corresponding power packages would then have the following characteristics:

Carbon Cathode Thickness	Carbon Volume	Watt Hours per Cell	Number of Cells	W.Hrs./Batteries
.574	73.62	368.1	12 X 6 = 72	26,503
.77	98.76	493.8	12 X 5 = 60	29,628
1.063	136.32	681.62	12 X 4 = 48	32,717
		W.Hrs./Cu.Ft. Cells	W.Hrs./Cu.Ft. Complete Package	
		4274	3276	
		4778	3662	
		5277	4044	

Battery Weight:

Previous available information has indicated that a #9 cell containing 44.7 in.³ of carbon cathode weighed 6.1 lbs. On this basis and using a weight of 100 lbs. for the super-insulation envelope and an additional 75 lb. for interconnection and terminations we arrive at the following estimated weights and energy density values:

Carbon Cathode Thickness	Weight of Power Package	W.Hrs./Lb.
.574	898 Lb.	29.5
.77	983 Lb.	30.14
1.063	1067 Lb.	30.66

Summary and Conclusions:

Further calculations have again indicated that a 25 KWH Carb-Tek[®] system power package can be designed into the available space existing in the battery compartment of a 2000 lb. MERDC fork-lift truck. It must be remembered that these conclusions are based on data generated in the design of the SOHIO #6 and #9 cell. For further refinement, new data is needed on present vintage cells.

Additionally it is recommended that engineering effort be expended on the following:

1. Interconnection of cells and cell modules.
2. Electrical terminations and connections to ambient temperature components.
3. Thermal termination and interface with ambient temperature components.
4. Establishment of firm battery operating parameters.

METRIC EQUIVALENTS TO UNITS IN APPENDIX I

Inch-pound units shown	Metric equivalent
<u>Page 1</u>	
3/4"	19 mm
7/8"	22 mm
8.09 cu. ft.	0.229 cu. m.
31.5 in x 19.625 x 22.655 = 8.09 cu.ft.	80.0 cm. x 49.8 cm. x 57.5 cm = 0.229 cu.m.
3090 w.hr./cu.ft	109,122 w.hr./cu.m.
5 W.hr./cu.in.	0.30 w.hr./cc
<u>Page 2</u>	
4" x 4 3/8" x .330" = 5.775 cu.in.	10.2 cm x 11.1 cm x 0.84 cm = 95.1 cc
.625" x 4 9/32" x 6" = 16.05 cu. in.	1.59 cm x 10.9 cm x 15.2 cm = 263.4 cc
3108.8 w.hr./cu.ft.	109,786 w.hr./cu.m.
7.203" x 7.203" x .875" = 45.397 cu.in.	18.30cm x 18.30cm x 2.22cm = 743.46cc
7 29/64" x 9 13/64" x 1 9/32" = 87.76 cu.in.	18.93cm x 23.38cm x 3.25cm = 1438.40cc
4469.34 w.hr./cu.in.	157,833 w.hr./cu.m.
5/8"	16 mm
1"	25 mm
1"	25 mm
29.5 in. x 17.625 in. x 20.625 in.	74.9 cm x 44.8 cm. x 52.4 cm
9 13/64 - 7 13/64 = 2.00 in.	23.38 cm - 18.30 cm = 5.08 cm
7 29/64 - 7 13/64 = .25 in.	18.93 cm - 18.30 cm = 0.63 cm
<u>Page 3</u>	
17.625"	44.77 cm
20.625"	52.39 cm
29.5"	74.93 cm
<u>Page 4</u>	
14.75"	37.47 cm
10.3125"	26.19 cm
12.75"	32.39 cm
10.06"	25.55 cm
5/8"	16 mm
80.165 cu.in.	1313.7 cc
3/4"	19 mm
96.198 cu.in.	1576.4 cc
7/8"	22 mm
112.23 cu.in.	1839.1 cc
1"	25 mm
128.26 cu.in	2101.8 cc
17.625"	44.77 cm
1.03 in.	2.62 cm
1.155 in.	2.93 cm
1.28"	3.25 cm
1.405"	3.57 cm
.979"	2.49 cm
.574"	1.46 cm
1.175"	2.98 cm
.77"	1.96 cm
1.465"	3.72 cm
1.063"	2.70 cm

(continued)

METRIC EQUIVALENTS TO UNITS IN APPENDIX J (continued)

Inch-pound units shown	Metric equivalent
Page 5	
.574"	1.46 cm
73.62 cu.in.	1206.4 cc
.77"	1.96 cm
98.76 cu.in.	1618.4 cc
1.063"	2.70 cm
136.32 cu.in.	2233.9 cc
4274 w.hr./cu.ft.	150,935 w.hr./cu.m.
3276 w.hr./cu.ft.	115,691 w.hr./cu.m.
4778 w.hr./cu.ft.	168,733 w.hr./cu.m.
3662 w.hr./cu.ft.	129,322 w.hr./cu.m.
5277 w.hr./cu.ft.	186,355 w.hr./cu.m.
4044 w.hr./cu.ft.	142,812 w.hr./cu.m.
44.7 cu.in.	732.5 cc
6.1 lb.	2.77 kg
100 lb.	45.36 kg
75 lb.	34.02 kg
898 lb.	407.33 kg
29.5 w.hr./lb.	65.04 w.hr./kg
983 lb.	445.88 kg
30.14 w.hr./lb.	66.45 w.hr./kg
1067 lb.	483.98 kg
30.66 w.hr./lb.	67.59 w.hr./kg

APPENDIX II

CONTRACT NO. DAAK02-73-C-0493

PRODUCTION & ENGINEERING METHODS FOR CARB-TEK^R BATTERIES IN FORK LIFT TRUCKS

APPENDIX TESTING REPORT - 31 AUGUST 1974

The independent QC testing of Carb-Tek^R cells has been planned to be performed at the ESB Technology Center, Yardley, Pa.

EQUIPMENT

Equipment secured and on order for this testing program is as follows:

1. Furnace:	Gruenberg Electric Co.
(on hand)	Model L120N100
	Serial No. 5288
	208 V - 4000 watts

Temp. Controller:	LOVE #52-518
(on order)	0-800°C

TEST CELLS

Two cells were secured for testing. These are SOHIO cell numbers 0436 and 0454.

TEST PLAN

Cells are to be heated to operating temperature (430-440°C) and inspected for bulging. They will then be temperature cycled by reducing to 100°C and reheating to operating temperature. If no cracks or leaks develop they will be given 5 cycles as shown in Table I to verify results reported by SOHIO.

TABLE I

Charge at 12A to 3.20 volts/cell - taper to 3.32 V
Float at 3.20 volts/cell for 16 hours
Discharge at 6.A to 1.0 volts/cell cutoff

After the above cycles the cells will be tested according to the modified MERDC discharge profile as listed in Table II. This profile has been scaled down from the MERDC profile which provided for constant power pulses from a 36 V, 720 Ahr battery. Our testing will be at constant current on a 2.75 V, 9 Ahr cell.

(continued)

APPENDIX II

(continued)

TABLE II

<u>Step No.</u>	<u>Time (sec.)</u>	<u>Amperes</u>
1	7	0.909
2	2	0.
3	8	0.932
4	2	0.
5	10	1.386
6	12	0.
7	10	1.045
8	2	0.
9	12	1.545
10	2	0.
11	10	1.386
12	12	0.
13	9	0.909
14	2	0.
15	8	0.932
16	2	0.
17	10	1.386
18	12	0.
19	15	1.045
20	5	0.945
21	3	1.045
22	5	0.
23	10	1.045
24	2	0.
25	6	1.773
26	2	0.

Repeat above profile 120 times (6 hours total).

Recharge at 5 amperes to 3.20 V or 1 hr. maximum.

Continue cycling until cell fa-ils to maintain greater than 1.5 volts on any discharge pulse.

TEST RESULTS

No cells have been tested to date due to lack of delivery of required equipment. When all necessary equipment has been received and installed, testing will proceed according to plan described.